

APPENDIX D

Quality Assurance Project Plan (Geomatrix 2007)

Quality Assurance Project Plan

Former Pechiney Cast Plate, Inc., Facility 3200 Fruitland Avenue, Vernon, California

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July 20, 2007

Project No. 10627.003.0





QUALITY ASSURANCE PROJECT PLAN FORMER PECHINEY CAST PLATE, INC., FACILITY VERNON, CALIFORNIA

July 20, 2007

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LIST OF ACRONYMS

COPC Chemical of Potential Concern

DI deionized

DQOs data quality objectives
EDD electronic data deliverable
ERB equipment rinsate blank

FS Feasibility Study

H&EC Health & Environmental Control (City of Vernon)

H&S health and safety

LCS laboratory control standards

MS matrix spike

MSD matrix spike duplicate

PARCCS precision, accuracy, representativeness, comparability, completeness,

and sensitivity

PCBs polychlorinated biphenyls

PE Professional Engineer (California State)
PG Professional Geologist (California State)

PIC Principal In Charge PM Project Manager

QAPP Quality Assurance Project Plan

QA Quality Assurance
QC Quality Control
RAP Remedial Action Plan

RL reporting limit

RPD relative percent difference
SOP standard operating procedure
SVOCs semi-volatile organic compounds
TPH total petroleum hydrocarbons

U.S. EPA United States Environmental Protection Agency

VOCs volatile organic compounds



QUALITY ASSURANCE PROJECT PLAN

Former Pechiney Cast Plate, Inc., Facility 3200 Fruitland Avenue, Vernon, California

1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) has been prepared by Geomatrix Consultants, Inc. (Geomatrix), on behalf of Pechiney Cast Plate, Inc. (Pechiney), to describe the quality assurance/quality control (QA/QC) procedures to be implemented during soil, soil vapor, and concrete sampling and perimeter air monitoring conducted during below-grade demolition and remediation activities at the former Pechiney Cast Plate, Inc. facility (Site) located at 3200 Fruitland Avenue, Vernon, California (Figure 1). As part of the demolition and remediation activities, soil and concrete samples will be collected and analyzed to provide confirmation of achieving soil and concrete remediation goals and to further any characterize impacted soil or concrete that may be discovered during removal of below-grade structures. In addition, site perimeter air monitoring samples will be collected and analyzed during various phases of below-grade demolition and excavation activities. Procedures and guidelines described in this document are not applicable to the collection and analysis of samples used for waste profiling purposes, however, these samples will be analyzed using the test methods described in this QAPP.

This QAPP presents the organization, objectives, planned activities, and specific QA/QC procedures. Specific protocols for sampling, sample handling and storage, chain of custody documentation, and laboratory and field analyses are described. This QAPP is written using the United States Environmental Protection Agency (U.S. EPA) guidelines (U.S. EPA, 2001, 2002b; U.S. EPA Region 9, 1989, 1997). This QAPP will be used in conjunction with the Feasibility Study/Remedial Action Plan (FS/RAP) (Geomatrix, 2007), Below Grade Demolition Plan (Geomatrix, 2006), below-grade demolition construction technical, and other related documents for this Project.

2.0 PROJECT MANAGEMENT

This section provides an overall approach to managing the work and addresses:

• project organization, roles, and responsibilities;



- problem definition;
- problem description;
- project Data Quality Objectives (DQOs) and criteria for measurement data;
- special training requirements or certificates required for work performed; and
- documentation and records management.

2.1 PROJECT ORGANIZATION AND ROLES AND RESPONSIBILITIES

The responsibilities of key project personnel are presented in this section and are presented on Figure 2. The QAPP will be distributed to all key project personnel.

2.1.1 City of Vernon Health & Environmental Control

The City of Vernon Health & Environmental Control (H&EC) project manager, Leonard Grossberg, has the regulatory oversight responsibility for this Project.

2.1.2 Client Representative

Mr. Greg Sutherland is representing the client, Pechiney. Mr. Sutherland is responsible for overseeing this Project as the client representative. Mr. Sutherland will work with the Geomatrix Project Manager (PM) to ensure that the project objectives and requirements are addressed.

2.1.3 Geomatrix Consultants, Inc.

Geomatrix is contracted to Pechiney to provide environmental consulting services for this Project. The following subsections describe the project organization and duties of the Geomatrix personnel assigned to the Project.

2.1.3.1 Principal in Charge, Principal Engineer

The Principal in Charge (PIC), Calvin H. Hardcastle, PE, is responsible for reviewing all technical aspects of the Project to ensure that all work elements meet the project objectives and technical standards, and are completed in accordance with the QAPP protocols. The PIC is provided technical information by the PM and the Task Leaders, quality assurance documentation by the QA Advisor, and health and safety information by the Project Health and Safety (H&S) Officer.



2.1.3.2 Project Manager

The PM, Linda Conlan, PG, is responsible for the scope, cost, and technical considerations related to the Project; staff and project coordination; and implementation of overall project QA/QC protocols related to the collection, completeness, and presentation of data. The PM oversees the technical work conducted by the Project Engineer and Task Leaders, quality assurance activities by the QA Advisor, and health and safety activities by the Project H&S Officer. The PM coordinates with the City of Vernon H&EC.

For the purposes of this QAPP, "PM" refers to the Geomatrix Project Manager.

2.1.3.3 Project Engineer

The Project Engineer, Bryan Stone, PE, is responsible for overseeing the Project and working with the Project Manger, QA Advisor, and Task Leaders during implementation of field activities. The Project Engineer will also oversee and coordinate, as necessary, field contractors involved with actual implementation of work, and supervise the Task Leader.

2.1.3.4 Quality Assurance Advisor

The QA Advisor, Margaret K. (Peggy) Peischl, PE, is responsible for reviewing the project QA program as it relates to the collection and completeness of data from field and laboratory operations, including training personnel to follow established protocols and procedures, and updating the QAPP as necessary.

2.1.3.5 Project Health and Safety Officer

The Project H&S Officer, Brian Swenson, PE, is responsible for developing, implementing, and updating the site-specific health and safety plan to be consistent with foreseeable conditions that may be encountered during field operations.

2.1.3.6 Task Leader

Brian Swenson, PE, will be assigned as the project's Task Leader responsible for executing the planned work elements, issuing specific instructions for performing assigned work elements, and ensuring that work is conducted in compliance with project-specific objectives and applicable QA procedures. The Task Leader will coordinate with the PM, Project Engineer, and QA Advisor to review general work plans and specific work elements. The Task Leader maintains all documentation and deliverables in the project files during the performance of the assigned tasks. For field sampling activities, the Task Leader will be responsible for



performing or overseeing the field work, preparing proper documentation, and sample handling for all on-site sampling activities.

2.1.4 Analytical Laboratory

A number of analytical laboratories will support this Project, and include:

- American Analytics of Chatsworth, California, will provide analytical services for the analysis of soil for total petroleum hydrocarbons (TPH), volatile organic compounds (VOCs), polychlorinated biphenyls (PCBs), metals, and semi-volatile organic compounds (SVOCs) and the analysis of soil vapor for VOCs. American Analytics will also provide analytical services for the analysis of concrete samples for PCBs;
- Air Toxics of Folsom, California, will provide analytical services for the analysis of the perimeter air monitoring samples for PCBs;
- Calscience of Garden Grove, California, will provide analytical services for the analysis of perimeter air monitoring samples for VOCs; and
- Chester LabNet of Tigard, Oregon, will provide analytical services for the analysis of perimeter air monitoring samples for PM-10 particulate.

The Laboratory Project Managers will be the primary laboratory contacts for the Task Leader and QA Advisor. The Quality Assurance Manual for each laboratory is provided in Appendix A.

2.2 PROJECT DEFINITION/BACKGROUND

The Site is comprised of approximately 26.9 acres and was formerly occupied by approximately 600,000 square feet of building area and was used to manufacture high-precision cast aluminum plates. Previous remedial investigations and assessments identified the following chemicals of potential concern (COPCs) at the Site:

- TPH, including Stoddard solvent;
- PCBs;
- VOCs;
- metals; and
- SVOCs.



The Site is presently zoned for industrial use. The City of Vernon is in the process of purchasing the property. The future site use will remain industrial, with the north portion of the Site anticipated for use as a power plant.

Site-specific remediation goals have been established for the Site and are briefly discussed in Section 2.3.2 and discussed in detail in the FS/RAP (Geomatrix, 2007). Remediation will be conducted in areas of the Site where soil and concrete concentrations exceed the site-specific remediation goals. The proposed remedial alternatives and remedy selection process for the remediation activities are discussed in detail in the FS/RAP. The locations of the proposed remedial action areas are included in the FS/RAP.

2.3 PROJECT DESCRIPTION

This project involves the following tasks:

- collection and analysis of soil and concrete samples for characterization of the extent of impacted soil discovered during demolition;
- collection of concrete samples for characterization of the extent of PCB impacts in concrete slabs;
- collection and analysis of soil vapor samples for further characterization or closure documentation:
- collection and analysis of soil samples to confirm the removal of impacted soil above the remediation goals;
- collection and analysis of investigative derived waste samples as required by the receiving disposal facilities; and
- quantification of potential air emissions related to demolition and remediation activities.

Sampling locations and rationale are discussed in the Soil, Soil Vapor, and Concrete Sampling and Analysis Plan (Appendix B) and the perimeter air sampling locations and rationale are discussed in the Perimeter Air Sampling Plan (Appendix C).

2.3.1 Measurements

A Soil, Soil Vapor, and Concrete Sampling and Analysis Plan (Appendix B) prepared for this project describes the procedures and protocols for soil and concrete sampling. A Perimeter Air Sampling Plan (Appendix C) describes the procedures and protocols for the perimeter air monitoring. The chemicals that will be analyzed are shown in Table 1. The primary target



analytes for this Project are trichloroethene, tetrachloroethene, PCBs, metals, and TPH for the gasoline range (C6-C12), the diesel range (C12-C22), the heavier hydrocarbon range (C22-C44), and Stoddard solvent. The following types of measurements may be collected:

- analytical results of soil, soil vapor, and concrete samples;
- analytical results of air monitoring samples;
- PM-10 dust monitoring; and
- meteorological data (wind direction, wind speed, temperature, relative humidity, and barometric pressure).

2.3.2 Site-Specific Remediation Goals

Site-specific remediation goals developed for the Site are presented in the FS/RAP. These goals were established using regulatory guidance, a human health risk assessment, and a groundwater attenuation model. Laboratory reporting limits (RLs) are less than the site-specific remediation goals in order to ensure that the goals are reached. The laboratory RLs for soil, soil vapor, and concrete are included with the Laboratory Quality Manual (Appendix A), and the laboratory RLs for perimeter air sampling are included in the Perimeter Air Sampling Plan (Appendix C).

2.3.3 Special Equipment and Personnel Requirements

The PM is responsible for ensuring that personnel will be trained to work and/or take measurements and samples as described in the Soil, Soil Vapor, and Concrete Sampling and Analysis and Perimeter Air Sampling Plans.

2.3.4 Assessment Techniques

Assessment activities required for the anticipated work are summarized as follows.

- Assessment of field operations: To evaluate the performance of field operations, sample collection documentation, chain of custody forms, and field notes and measurements will be reviewed. Unannounced field audits may be conducted.
- Assessment of laboratory operations: The selected analytical laboratories have internal audit programs that are applied to assess the degree of adherence to the policies and procedures found in their Quality Assurance Manual. Additionally, for each individual task, the PM and/or Task Leader will be in frequent contact with the analytical laboratory to assess progress in meeting DQOs and to identify problems requiring corrective action.



Specific details of assessment procedures can be found in Section 4.0.

2.3.5 Work Schedule

The anticipated schedule for this project is summarized on Figure 3.

2.3.6 Project and Quality Records and Reports

Critical records for the work include:

- Daily Field Records;
- Chain of Custody Records;
- Sample Control Logs;
- laboratory reports; and
- A summary report of the results.

More details on project records and reports can be found in Section 2.7.

2.4 DATA QUALITY OBJECTIVES

Data collected on a site needs to be of sufficient quality and quantity to support defensible decision making. DQOs ascertain the type, quality, and quantity of data necessary to address the "problem" before the sampling and analysis begin. The U.S. EPA guidance document, QA-G4 (U.S. EPA, 2000) outlines a seven-step process for establishing DQOs to address the "problem" (the "problem" refers to the sampling activities associated with below-grade demolition and remediation). These steps are as follows.

- 1. **State the Problem.** Concisely describe the "problem" to be studied.
- 2. **Identify the Decision.** Identify the decision that will solve the "problem" using data
- 3. **Identify the Inputs to the Decision.** Identify the information needed and the resulting measurements that need to be made in order to support the decision
- 4. **Define the Study Boundaries.** Specify the conditions (time periods, spatial areas, and situations) to which the decision will apply and within which the data will be collected.
- 5. **Develop a Decision Rule.** Define the conditions by which the decision-maker will choose among alternative risk management actions. This is usually specified in the form of an "if…then…" statement.



- 6. **Specify Acceptable Limits on Decision Errors.** Define in statistical terms the decision-maker's acceptable error rate based on the consequence of making an incorrect decision.
- 7. **Optimize the Sampling Design.** Evaluate the results of the previous steps and develop the most resource-efficient design for data collection that meets all of the DQOs.

DQO Step 1: Problem Statement

The following "problem" statements are relevant to the soil, soil vapor, concrete, and perimeter air sampling activities for the Site:

- 1. The concentrations of COPCs in soil and concrete following the remediation and excavation activities need to meet the site-specific remediation goals that were developed based on potential future exposure scenarios (protection of human health and groundwater).
- 2. The concentration of VOCs in soil vapor for further characterization (e.g., in the Stoddard solvent impacted areas) or following the remediation activities as needed to meet site-specific criteria.
- 3. Potential air emissions due to and during demolition and remediation activities need to be assessed at the perimeter of the Site.

DQO Step 2: Identify the Decision

The following decisions and related inquiries are required for each "problem" statement.

- 1. The concentrations of COPCs in soil and concrete following the remediation and excavation activities need to meet the site-specific remediation goals that were developed based on potential future exposure scenarios (protection of human health and groundwater).
 - a. What are the concentrations of COPCs in soil at the perimeter of the excavations?
- 2. The concentration of VOCs in soil vapor for further characterization (e.g., in the Stoddard solvent impacted areas) or following the remediation activities as needed to meet site-specific criteria.
 - b. What are the concentrations of target VOCs in soil vapor (e.g., Stoddard solvent impacted areas)?
 - c. What are the remaining concentrations of VOCs in soil vapor following remediation?



- 3. Potential air emissions due to and during demolition and remediation activities need to be assessed at the perimeter of the Site.
 - d. What are the concentrations of air emissions at the Site perimeter during demolition and remediation activities?

DQO Step 3: Inputs to the Decisions

- 1. The concentrations of COPCs in soil and concrete following the remediation and excavation activities need to meet the site-specific remediation goals that were developed based on potential future exposure scenarios (protection of human health and groundwater).
 - a. Collect confirmation soil samples along the perimeter of the extents of excavation and at potential impacted areas discovered during demolition.
- 2. The concentration of VOCs in soil vapor for further characterization (e.g., in the Stoddard solvent impacted areas) or following the remediation activities as needed to meet site-specific criteria.
 - a. Collect confirmation soil vapor samples within the Stoddard solvent areas (e.g., Phase IV or Building 112A) to assess potential risk to future receptors (indoor air pathway).
 - b. Collect confirmation soil vapor and soil samples during and following remediation (e.g., soil vapor extraction/bioventing).
- 3. Potential air emissions due to and during demolition and remediation activities need to be assessed at the perimeter of the Site.
 - a. Collect air and dust samples along the Site perimeter during demolition and excavation activities.

DQO Step 4: Study Boundaries

Previous investigations at the site indicate the areas of impacted soil. However, other impacted soil may be encountered during the demolition and remediation activities. The potential vertical extent of the remediation and excavation activities is from approximately ground surface to 15 feet below ground surface.

DQO Step 5: Decision Rules

The decision rules for the "problem" statement are as follows:

1. The concentrations of COPCs in soil and concrete following the remediation and excavation activities need to meet the site-specific remediation goals that were



developed based on potential future exposure scenarios (protection of human health and the environment).

- a. If the results of initial confirmation soil sampling indicate the presence of COPCs in soil above site-specific remediation goals (Section 2.3.2), then further remediation and excavation will be necessary. Further evaluation may include additional confirmation sampling, extending the spatial limit of the work area, or other appropriate assessment activities.
- b. If the results of the confirmation soil sampling are less than or equal to sitespecific remediation goals in soil, then no further remediation or excavation activities will be performed.
- 2. The concentration of VOCs in soil vapor for further characterization (e.g., in the Stoddard solvent impacted areas) or following the remediation activities as needed to meet site-specific criteria.
 - a. If the results of the characterization sampling are above risk-based levels for potential indoor air exposure, remediation may be implemented.
 - b. If the results of the characterization sampling are below risk-based levels for indoor air exposure, no further action will be necessary.
- 3. Potential air emissions due to and during demolition and remediation activities need to be assessed at the perimeter of the Site.
 - a. If the results of perimeter air sampling exceed the action levels established in the Perimeter Air Sampling Plan, then appropriate action will be conducted as outlined in the Plan.
 - b. If the results of perimeter air sampling are less than or equal to action levels, then no further action will be required.

DQO Step 6: Limits on Decision Error

A decision error occurs when the data are misleading, and as a result, the wrong decision is made. The possibility of a decision error exists because the parameter of interest is estimated using the data.

Variability introduced by sampling and analysis of COPCs in soil could result in a conclusion that the COPCs are present at concentrations greater than the site-specific remediation goals for soil remaining at the Site. The consequence of incorrectly deciding that a COPC concentration exceeds its respective remediation goal is unnecessary additional work, including additional soil excavation, sampling, analysis or other assessment work. The consequence of incorrectly



deciding that a COPC concentration does not exceed its respective remediation goal is that impacted soil would not be excavated.

For air and dust sampling, variability could result in a conclusion that COPCs are present in concentrations greater than the action levels at the perimeter of the Site. The consequence of incorrectly deciding that a COPC concentration exceeds its respective action level is unnecessary additional work, including additional vapor or dust mitigation measures during excavation activities, sampling, or analysis. The consequence of incorrectly deciding that a COPC concentration does not exceed its respective action level at the Site perimeter is air emissions or dust with elevated COPC concentrations are leaving the Site.

Data variability and, therefore, the probability of a decision error during below-grade demolition and remediation activities will be reduced by collecting an appropriate number of samples using experienced personnel and performing the analyses using approved analytical methods. Details of the sampling approach are presented in Appendix B.

DQO Step 7: Sample Design Optimization

Sampling locations, number of samples, and analytical methodologies are proposed for soil, soil vapor, concrete sampling, and perimeter air sampling are included in Appendix B and C, respectively. Additional sampling may be warranted based on the findings as work progresses during remediation activities. Any modifications to the sampling plans will be spelled out in the reports of findings.

2.5 METHOD PERFORMANCE OBJECTIVES

Analytical performance requirements for work performed are expressed in terms of precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS). The following subsections present a summary of each PARCCS parameter and calculation equations as appropriate. A summary of the PARCCS parameters, frequency, and acceptance criteria are included in Table 2.

2.5.1 Precision

Precision is a measurement of the degree of agreement of replicate data, which is quantitatively assessed based on the relative percent difference (RPD) or standard deviation.



Field Precision

Field precision is typically assessed through the collection and measurement field duplicate samples. Field duplicates will be collected for soil vapor characterization testing. Duplicate samples for other matrices (e.g., soil, concrete, etc.) are not anticipated for this project based on the media to be tested.

Laboratory Precision

Laboratory precision accuracy is assessed by calculating RPDs for two replicate samples. The precision of the analysis can be inferred through one of the following: laboratory control stardards (LCS) and laboratory control duplicate samples; matrix spike (MS) and matrix spike duplicate (MSD) samples, or unspiked duplicate samples. The laboratory analyzes one or more of these duplicate samples at a rate of one per batch of 20 samples per matrix.

The MS/MSD samples provide information about the effect of the sample matrix on extraction and measurement methodology. An MS/MSD pair will be analyzed at a rate of one per batch of 20 or fewer investigative samples per matrix.

The precision of laboratory analyses will be assessed by calculating the RPD for each pair of duplicate samples (MS/MSD), laboratory control sample spike duplicates, unspiked duplicate samples, and field duplicate sets using the following equation:

$$\% RPD = \frac{S_1 - S_2}{S_{av}} \times 100$$

where:

 S_1 = first sample result (original or MS value)

 S_2 = second sample result (duplicate or MSD value)

 S_{av} = average of sample and duplicate = $(S_1 + S_2)/2$

2.5.2 Accuracy

Accuracy is the degree of agreement between a measurement or observation and an accepted value.

Field Accuracy

Field accuracy, assessed through appropriate field equipment and trip blanks, is achieved by adhering to all sampling, handling, preservation, and holding time requirements. Field blank



samples are analyzed to check for possible procedural contamination that could affect samples. Equipment rinse blanks are used to assess the adequacy of decontamination of sampling equipment between individual sample collections. Trip blanks are used to assess the potential for contamination of samples due to migration of contaminants (e.g., VOCs) during sample shipment, handling, and/or storage. Accuracy of field instruments is assessed by daily instrument calibration and calibration checks.

Laboratory Accuracy

Laboratory accuracy is assessed by analyzing matrix spikes and LCS. The results are expressed as a percent recovery. Surrogate recoveries may also be used to assess accuracy. Method blanks are used to assess possible contamination from laboratory procedures. Laboratory control samples, method blanks, and preparation blanks will be analyzed at least once with each analytical batch, with a minimum of one for every 20 samples. The percent recovery (percent R) is calculated with the following equation:

$$\% R = \frac{A - B}{C} \times 100$$

where:

A = The sample result

B = The background level determined by a separate analysis of the unspiked sample

C =The amount of the spike added

2.5.3 Representativeness

Representativeness is a qualitative measure of the degree to which sample data accurately and precisely represent a characteristic environmental condition. Representativeness is a subjective parameter used to evaluate the efficiency of the sampling plan design. Representativeness is demonstrated in the project planning documents by providing full descriptions of the sampling techniques and the rationale used for selecting sampling locations. The measure of representativeness is established during preparation of the sampling and analysis approach and rationale, and then reassessed during the data usability process. Numerical goals cannot be used to evaluate this subjective measure.



2.5.4 Completeness

Completeness is a measure of the quantity of valid data obtained from a measurement system compared to the quantity that was planned under normal conditions. Percent completeness is calculated with the following equation:

$$\% \ \textit{Completeness} \ = \frac{\textit{Valid Data Obtained}}{\textit{Total Data Planned}} \times 100$$

Experience on similar projects has shown that a reasonable goal, considering combined historical field and laboratory performance, is 90 percent completeness. If insufficient valid data are obtained, the PM will initiate corrective action.

2.5.5 Comparability

Comparability expresses the confidence with which one dataset can be compared with another dataset obtained during parallel or previous investigations. Comparability can be related to precision and accuracy because these parameters are measures of data reliability.

Chemical samples from the same media generally are considered comparable if the same procedures for collecting and analyzing the samples are used, if the samples comply with the same QA/QC procedures, and if the units of measurement are the same. To provide comparability, data generated will be subject to the QA/QC procedures specified in this QAPP, and the Soil, Soil Vapor, and Concrete Sampling and Analysis and Perimeter Air Sampling Plans.

2.5.6 Sensitivity

Sensitivity is the measure of the concentration at which an analytical method can positively identify and report analytical results. The sensitivity of a given method commonly is referred to as the detection limit. RL is the concentration of the target analyte that the laboratory has demonstrated the ability to measure within specified limits of precision and accuracy during routine laboratory operating conditions. This value is variable and highly matrix-dependent. It is the minimum concentration that the laboratory will report as unqualified. For sensitivity, the quality objective is to analyze data using a method that achieves RLs that are below or equal to the task-specific remedial goals or concentrations. The RLs for analytes anticipated for this work are presented in Table 1 and in Appendix A.



2.6 SPECIAL TRAINING, REQUIREMENTS, AND CERTIFICATION

The PM is responsible for assembling a project team having the necessary experience and technical skills to conduct the work. Part of the process is to identify special training requirements or certifications necessary to successfully execute the project. Technical documents will require the signature of a Professional Geologist, Civil Engineer, Environmental Engineer, or qualified environmental professional. Additionally, all field personnel will have the appropriate health and safety training. No other specialized training or certifications are anticipated for this work.

2.7 DOCUMENTATION AND RECORDS

This section identifies critical field and laboratory records required for most sampling work, information to be included in reports, the format for reporting data in analytical data report packages, and the document control procedures to be used.

2.7.1 Required Records

Records required for the project include field and laboratory records, and technical reports. Field records are described in Section 3.1.1 of this QAPP. Laboratory records are described in Section 2.7.2.

2.7.2 Laboratory Records

Analytical results will be reported in the laboratory's approved format described below. In addition to the reported data, the laboratory data report will, at a minimum, include a narrative that will discuss any problems or discrepancies, and sufficient calibration and QC information to determine that the method was within control limits at the time that the samples were analyzed. Laboratory records will include the following:

- case narrative;
- chain of custody documentation (external);
- final analyte concentration including RL, laboratory qualifiers, and re-analyses;
- laboratory sample identification (ID), field sample ID, matrix, and dilution factors;
- sample collection receipt, extraction, and analysis dates for holding time verification;
- percent recovery of each surrogate (as appropriate);



- surrogate recovery control limits;
- percent recovery of each compound in the MS sample (as appropriate);
- MS recovery control limits (as appropriate);
- RPD for all MS/MSD results (as appropriate);
- RPD control limits for MS/MSD reports (as appropriate);
- LCS results when analyzed;
- recovery control limits for LCS;
- condition and temperature of samples upon receipt;
- results for method blanks, field blanks, equipment blanks, and trip blanks; and
- method blank summary indicating associated samples.

In addition to the hard-copy report requirements, the laboratory will provide electronic data deliverables (EDDs) conforming to an American Standard Code for Information Interchange comma-delimited, Microsoft Excel, or EarthSoft, Inc., EQuIS format, as specified, for all data reported. The standard laboratory turnaround time will be 10 working days.

The laboratory's internal records management protocols are described in their Quality Assurance Plans.

2.7.3 Records Maintenance and Storage

Documents relating to the Project will be controlled to provide proper distribution, filing, and retrieval, and to assure that revisions are properly recorded, distributed, and filed. Project records will be stored and maintained by Geomatrix staff. The PM is responsible for organizing, storing, and cataloging project information. The PM also is responsible for collecting records and supporting data from project team members. Once cataloged, project records are filed by category in the appropriate project file. Filed documents are available to Geomatrix staff through checkout procedures developed to protect the integrity of project files. Individual project team members may maintain separate files or notebooks for individual tasks. Additional information on records management can be found in Section 3.10 of this QAPP.



3.0 MEASUREMENT AND DATA ACQUISITION

This section describes the design and implementation of measurement procedures and discusses the methods to be used for sampling, analysis, data handling, and QC in support of the tasks performed. The following specific aspects of measurement and data acquisition will be covered in this section:

- design of sampling process;
- requirements for sampling methods;
- requirements for sample handling and custody;
- requirements for analytical methods;
- QC requirements;
- requirements for instrument/equipment testing, inspection, and maintenance;
- instrument calibration and frequency;
- requirements for inspection and acceptance of supplies and consumables;
- requirements for data acquisition; and
- data management.

3.1 SAMPLING PROCESS DESIGN

The planned sampling locations and rationale for their selection is discussed in the Soil, Soil Vapor, and Concrete Sampling and Analysis and Perimeter Air Sampling Plans (Appendix B and C, respectively), and the analytical parameters are shown in Table 1.

3.1.1 Field Sampling Documentation

The Task Leader and other field sampling team members will maintain field notes to provide a daily record of significant events, observations, and measurements collected during sampling. Information pertinent to sampling will be recorded in the field notes or on activity-specific data forms. Each day's field note entries will be signed and dated and will include:

- date and time of entry, and weather and environmental conditions during the field activity;
- project name and number;



- location of sampling activity;
- name of field crew members;
- name of site visitors;
- sample media (e.g., soil, soil vapor, concrete, or air);
- sample collection method (e.g., summa canister, direct push probe, grab, drive sample, or backhoe bucket, automated air sampler); and
- number of samples taken.

When activity-specific data forms are used, they will also include:

- investigation location;
- sampler's initials;
- sampling medium; and
- sampling method.

The following information will be recorded either in the field notes or on the activity-specific data forms:

- volume and number of samples taken;
- date and time of collection;
- sample depth;
- sample identification number(s), including well name and/or number;
- sample destination (e.g., laboratory);
- field observations;
- field measurements; and
- sample handling (preservation).

Selected field notes and sampling forms are provided in Appendix D.

Original data recorded in the field notes, field data forms, sample labels, and chain of custody forms must be written using waterproof, indelible ink. None of these documents are to be



destroyed or discarded, even if one is illegible or contains inaccuracies requiring document replacement. If an error is made on an accountable document assigned to one individual, that individual will make all corrections simply by crossing a line through the error, initialing and dating the correction, and entering the correct information. The erroneous information will not be obliterated. Any subsequent error discovered on an accountable document will be corrected by the person who made the entry.

3.1.2 Sample Identification

The method of sample identification used depends on the type of sample collected and the sample container type. The field data are recorded in field notes or activity-specific data sheets along with sample identity information while in the custody of the sampling team. A sample label will be completed and attached to each sample container for every sample collected. Labels consist of a waterproof material backed with a water-resistant adhesive. Labels are to be filled out using waterproof ink, and are to contain at least the following information:

- project name and number;
- sampling date and time;
- sample identification number (including well name and/or number);
- preservatives, if any;
- sampler's initials; and
- analyses to be conducted.

Each analytical sample will be assigned a unique number consisting of an alphanumeric code that identifies the investigative area, feature type (well, bore, etc.), the specific sampling location, phase of work, and depths (for discrete-depth samples). These numbers will be tracked from collection through laboratory analysis and into the final reports. The sample number will be cross-referenced with the site name and sample location on the chain of custody form. Additional sample volume will be collected for samples identified by the Task Leader for the laboratory QC (i.e., MS/MSD).

3.2 SAMPLING METHOD REQUIREMENTS

Sampling procedures, methods, and equipment anticipated for this work are described in this section. Decontamination procedures and corrective action procedures also are described.



3.2.1 Soil, Soil Vapor, Concrete, and Air Sampling Procedures

Sampling procedures, methods, and equipment anticipated for this work are described in the Soil, Soil Vapor, and Concrete Sampling and Analysis and Perimeter Air Sampling Plans. Departures from the procedures must be documented and approved by the PM.

A complete set of sampling containers will be prepared for each sample in advance of the sampling event. Containers will be labeled with the date, sample number, project name, sampler's name or initials, parameters for analysis (method numbers where possible), and type of preservation. The laboratory will prepare the sample containers and add preservatives, if appropriate, to the containers prior to shipment to the field sampling team.

3.2.2 Equipment Decontamination Procedures

Equipment decontamination procedures are intended to reduce the possibility of sample contamination and cross contamination between sampling points. Decontamination procedures are described in the Soil, Soil Vapor, and Concrete Sampling and Analysis and Perimeter Air Sampling Plans.

3.2.3 Support Facilities for Sampling Methods

American Analytics of Chatsworth, California, will provide analytical services for the soil, soil vapor, and concrete samples collected for this Project. Air Toxics of Folsom, California, Calscience of Garden Grove, California, and Chester LabNet of Tigard, Oregon, will provide analytical services for the air samples collected for this Project.

3.2.4 Sampling/Measurement System Failure Response and Corrective Action

If QC surveillance and/or field audits detect unacceptable conditions or data, the PM, in conjunction with the QA Advisor, will be responsible for developing and directing implementation of corrective actions. Corrective actions will include one or more of the following:

- identifying the source of the violation;
- evaluating and amending sampling and analytical procedures; and/or
- accepting data but flagging it to indicate the level of uncertainty associated with failure to meet the specified QC performance criteria.

Any finding that requires corrective action must be documented to the PM. The QA Advisor will check that corrective actions have been implemented and that the problem has been



resolved. If more easily addressed problems are encountered in the field or the laboratory, such problems will be addressed and the corrective action noted in the appropriate laboratory or field data form.

If an error is made on an accountable document assigned to one individual, that individual will make all corrections simply by crossing a line through the error, entering the correct information, and initialing and dating the correction. The erroneous information will not be obliterated. The person who made the entry will correct any subsequent error discovered on an accountable document.

3.2.5 Sample Equipment, Preservation, and Holding Time Requirements

The sample containers, preservative requirements, and maximum holding times for the analytical methods are presented in Table 1.

3.3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Proper sample handling, appropriate shipment, and maintenance of chain of custody records are key to building the documentation and support for data that can be used to make program decisions. It is essential that all sample handling and sample chain of custody requirements be met in a complete, accurate, and consistent manner. Requirements for sample handling and custody must be met for all samples collected.

3.3.1 Sample Custody

Sample custody and documentation procedures described herein must be followed throughout sample collection activities. Components of sample custody procedures include the use of field memoranda, sample labels, custody seals, and chain of custody forms. The chain of custody form must accompany the samples during shipment from the field to the laboratory.

A sample is under custody under the following conditions.

- it is in one's possession;
- it is in one's view after being in his or her physical possession;
- it was in one's physical possession and that person then locked it up to prevent tampering; and
- it is in a designated and identified secure area.



The following procedures must be used to document, establish, and maintain custody of field samples.

- A label will be completed and attached to each sample container for every sample
 collected. Labels consist of a waterproof material backed with a water-resistant
 adhesive. Labels are to be filled out using waterproof ink, making sure that they are
 legible and affixed firmly on the sample container. Sample labels are to contain at
 least the following information: project number; sampling date and time; sample
 identification number; investigation location; preservatives, if any; sampler's
 initials; and analyses to be conducted.
- All sample-related information must be recorded in the field notes or on activityspecific data forms.
- The field sampler must retain custody of samples until they are transferred or properly dispatched.
- To simplify the chain of custody record and reduce potential problems, as few
 people as possible should handle samples or physical evidence. For this reason, one
 individual from the field sampling team should be designated as the responsible
 individual for all sample transfer activities. This individual will be responsible for
 the care and custody of the samples until they are properly transferred to another
 person or facility.
- A chain of custody record shall accompany all samples. The chain of custody record documents the transfer of custody of samples from the field investigator to another person, the laboratory, or other organizational entities. Signatures that acknowledge relinquishment and receipt of the samples must accompany each change of possession. Chain of custody records will be prepared for groups of samples collected at a given location on a given day. A chain of custody form will accompany every shipment of samples to the laboratory. A copy of each chain of custody form will be made and retained in the project file.
- The chain of custody form makes provision for documenting sample integrity and the identity of persons involved in sample transfer. Information entered on the chain of custody form will consist of:
 - o project name and number;
 - o chain of custody form serial number;
 - o number of containers/samples;
 - o sample numbers;
 - o sampler/recorder's signature;



- o date and time of collection of each sample;
- o collection location;
- o sample type;
- o analyses requested;
- o inclusive dates of possession;
- o name of person receiving the sample;
- o date of receipt of sample; and
- o matrix spike and matrix spike duplicate (MS/MSD) samples (if requested).

Completed chain of custody forms will be inserted into a plastic cover and placed inside the container used to transport samples from the field to the laboratory. A copy of a typical chain of custody form to be used is included in Appendix D. When samples are relinquished to a shipping company for transport, the tracking number from the shipping bill will be recorded on the chain of custody form.

3.3.2 Laboratory Sample Handling and Custody

The Task Leader will notify the Laboratory Project Manager of upcoming field-sampling activities and the subsequent transfer of samples to the laboratory. This notification will include information concerning the number and type of samples to be shipped, analyses requested, and the expected date of arrival. The Laboratory Project Manager will notify appropriate laboratory personnel, including the sample custodian, about the expected shipment. Upon arrival at the laboratory, the samples will be received and logged in by a trained sample custodian in accordance with the laboratory's sample handling and internal custody program. Upon sample receipt, the sample custodian is responsible for performing the following activities where appropriate:

- examining the shipping containers to verify that custody tape is intact (if used);
- measuring and documenting the shipping container temperature;
- examining all sample containers for damage;
- comparing samples received against those listed on the chain of custody record;
- verifying that sample holding times have not been exceeded;



- analyzing sample temperatures and documenting any variations from the acceptable range;
- analyzing sample pH, if required, and documenting the pH;
- immediately signing and dating chain of custody record after accepting shipment;
- noting and documenting any sample receipt problems, initiating a Condition Upon Receipt report, and notifying the Laboratory Project Manager;
- attaching the laboratory's sample container labels with laboratory identification number and test; and
- placing the samples in proper laboratory storage.

The Laboratory Project Manager is responsible for contacting the PM as soon as possible if any problems are identified during sample receipt. All problems identified during sample receipt will be resolved prior to sample preparation and analysis.

Following sample receipt, the sample custodian is responsible for logging the samples in the laboratory log-in book and/or the Laboratory Information Management System with the following information:

- laboratory project number;
- sample numbers (laboratory and client);
- type of samples;
- required tests; and
- date received.

The sample custodian is also responsible for notifying the Laboratory Project Manager and appropriate Task Leader of sample arrival and for placing completed chain of custody records, waybills, and any additional documentation in the project file.

Samples will be stored appropriately within the laboratory to maintain any prescribed temperature, protect against contamination, and maintain the security of the samples.

Sample custody procedures within the laboratory will be followed to appropriately document the handling and possession of the sample from receipt until final analysis and disposal. If any samples are transferred to a different laboratory, the transfer will be done under chain of



custody procedures and the labs will maintain the appropriate documentation to preserve the traceability of the samples through final analysis and disposal.

3.3.3 Sample Packing and Shipping

Field personnel, laboratory courier, or commercial shipping services (such as UPS or Federal Express) will deliver samples to the designated laboratory. The method of shipment will be noted on the chain of custody form. During the field effort, the Task Leader or a designee will inform the laboratory daily of planned shipments. Hard plastic ice chests or coolers with similar durability will be used for shipping samples. The coolers must be able to withstand a 4-foot drop onto solid concrete in the position most likely to cause damage. The samples must be cushioned so as to sustain the least amount of damage if such a fall should occur. After packing is complete for UPS or Federal Express shipping, the cooler will be taped shut with chain of custody seals affixed across the top and bottom joints. Each container will be clearly marked with a sticker displaying the originator's address.

The following procedures must be used when transferring samples for shipment.

- A chain of custody record must accompany samples. When transferring possession
 of samples, the individuals relinquishing and receiving must sign, date, and note the
 time on the record. This record documents transfer of custody of samples from the
 field sampler to another person or to the laboratory. Overnight shipping companies
 will not be required to sign the chain of custody record. A copy of the receipt of
 shipment will accompany the chain of custody record.
- Samples must be properly packaged for shipment and dispatched to the appropriate laboratory for analysis with a signed chain of custody record for each shipment.
- A chain of custody record identifying the contents must accompany all shipments.
 The original record must accompany the shipment, and the Task Leader must retain a copy.
- A temperature blank will be included in each cooler.

3.4 ANALYTICAL METHODS REQUIREMENTS

This section describes the general requirements for analytical methods that may be performed, including preparation/extraction procedures where appropriate and method performance requirements. Laboratory analyses will be conducted by American Analytics, Air Toxics, Calscience, and Chester LabNet. As needed, the laboratories may send samples to other facilities. The laboratory's Quality Assurance Plans contain summary information from the analytical methods, including the following:



- sample containers, preservatives, and holding times;
- calibration requirements, including frequency and acceptance criteria;
- laboratory quality control samples, including frequency, acceptance criteria, and corrective actions; and
- method RLs.

More detailed information on the laboratory's analytical methods is contained in laboratory-specific standard operating procedures (SOPs) that can be obtained directly from the laboratory.

3.4.1 Analytical Methods

In general, all analyses will utilize EPA-approved methods or other recognized standard methods. Method references for laboratory analyses that will be performed for the anticipated work are provided in Table 1, including preparation/extraction methods where appropriate.

3.4.2 Reporting Limits

Laboratory-specific RLs are included with the laboratory Quality Assurance Plans (Appendix A) and the laboratory-specific RLs for the perimeter air samples are included in Table 1. The laboratory's RLs may be modified based on the laboratory's current performance, changes to the methods, and any method detection limit studies. RLs must be lower than the site-specific remediation goals outlined in Section 2.3.2.

The actual RLs reported by the laboratories will be evaluated in the DQO process for the proposed work. The adequacy of RLs is important DQOs because they are used to identify the nature and extent of chemical impacts as well as the risk due to potential exposure. In general, the RLs for the various analytical methods reported by the laboratory appear to be sufficient for the anticipated use of data. In the event a task-specific target is less than the RLs reported by the laboratory, a discussion of the exception and any recommended solutions will be presented in the associated Final Report.

3.4.3 Laboratory Method Performance Requirements

A description of the method-specific QC samples that the laboratories use are provided in their Quality Assurance Plans, including the types of QC samples to be run, frequency, acceptance criteria, and corrective action to be taken when acceptance criteria are not met. The laboratory



analyst will review results of the QC samples against the acceptance criteria. Any identified discrepancies will trigger the laboratory's internal corrective action system as described below.

3.4.4 Laboratory Corrective Action

The laboratories have a formal corrective action system in place to provide that prompt action is taken when an unplanned deviation from a procedure or plan occurs and that, whenever possible, corrective actions include measures to prevent the reoccurrence of deviations. Specific corrective actions to be taken when a QC sample does not meet acceptance criteria are presented in the laboratory's Quality Assurance Plans. The following is a description of how information from the laboratory's corrective action system is communicated to the project team.

Each laboratory's corrective action procedure includes promptly notifying the project contact of any significant problems or discrepancies. The Laboratory Project Manager is responsible for reporting to the PM or other identified project contact any significant problems or discrepancies that occur as analyses are conducted. The Laboratory Project Manager is also responsible for assuring that corrective action is taken where appropriate to prevent the reoccurrence of similar problems or discrepancies. In addition, each analytical data report will include a case narrative that discusses any problems or discrepancies, and sufficient calibration and QC information to verify that the method was in control at the time the samples were analyzed. The case narrative will include a discussion of any corrective action taken by the laboratory to prevent the reoccurrence of similar problems or discrepancies.

3.5 QUALITY CONTROL REQUIREMENTS

This section presents the field QC checks that will be performed during field investigations, including a discussion of field QC samples with frequency and acceptance criteria and field corrective action procedures. A discussion of laboratory QC samples and laboratory corrective action was presented in the previous section (Section 3.4).

3.5.1 Field QC Samples

Typically, field contamination is assessed through the collection of different types of blank samples. Equipment rinsate blank (ERB) samples are obtained by passing distilled or deionized (DI) water, as appropriate, over or through the decontaminated reusable equipment used for sampling. These blank samples provide the best overall means of assessing contamination arising from equipment, ambient conditions, sample containers, transit, and the laboratory.



Temperature blanks will be placed in each cooler shipped to the laboratory. Temperature blanks will be provided by the laboratory and will be used to obtain a representative temperature of the cooler upon laboratory receipt the cooler containing samples.

Trip blank samples are prepared by the laboratory and shipped to and from the field. These blank samples help assess contamination from the laboratory, the shipping process, and are only for VOCs.

Definitions for these types of samples are provided in the following subsections. The specific field QC samples required for the anticipated sampling program is presented in Table 3.

3.5.1.1 Equipment Rinsate Blank Samples

Equipment rinsate blank samples are used to monitor effectiveness of the decontamination process. ERBs contain DI water passed through and over the surface of decontaminated reusable sampling equipment. The rinse water is collected in sample bottles, preserved as necessary, and handled in the same manner as the samples. The ERBs will be analyzed for the same analytes as the corresponding samples collected that day.

3.5.1.2 Field Decontamination Water Blanks

Field blanks are samples of the source water used for decontamination and steam cleaning. This blank is used to monitor for potential contaminants introduced from the water source during field decontamination procedures. Typically, at least one sample for each source of water or one field blank of analyte-free water for a specified event will be collected and analyzed for the same parameters as the corresponding field environmental samples. If more than one source of DI water is used, or if potable water from more than one location is used, additional field blanks are collected because these constitute different sources. The requirement for field blanks will be at the discretion of the PM and presented in the Soil Sampling and Analysis and Perimeter Air Sampling Plans.

3.5.1.3 Trip Blanks

Trip blanks are used to detect VOC contamination during sample shipping and handling. Trip blanks are 40-milliliter volatile organic analysis vials of water that are filled by the laboratory, transported to the sampling site, and returned to the laboratory with VOC samples. Trip blanks are not opened in the field. The planned frequency for trip blanks is one trip blank per cooler containing samples for VOC analysis.



3.5.1.4 Temperature Blank

Temperature blanks are used to provide a representative temperature of the cooler containing samples upon laboratory delivery. Temperature blanks are provided by the laboratory before sampling occurs.

3.5.2 Field Corrective Action

Problems that require corrective action may be encountered in the field. Any finding that requires corrective action must be documented to the PM. The Project QA Officer will confirm that corrective actions have been implemented and that the problem has been resolved. If more easily addressed problems are encountered in the field, such problems will be addressed and the corrective action noted in the appropriate field memoranda. If an error is made on an accountable document assigned to one individual, that individual will make all corrections by crossing a line through the error, entering the correct information, and initialing and dating the correction. The erroneous information will not be obliterated. The person who made the entry will correct any subsequent error discovered on an accountable document.

3.6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

Maintenance and inspection of both field and laboratory equipment are described in the following subsections.

3.6.1 Field Instrument/Equipment

Preventative maintenance of field measurement instrumentation and equipment will be performed according to the SOPs presented in the manufacturer's instructions. The field staff is responsible for providing that all instrumentation is operating properly prior to use. If problems are encountered, they will be documented in a field notes.

3.6.2 Laboratory Instrument/Equipment

Testing, inspection, and maintenance of laboratory instruments/equipment will be conducted in accordance with the procedures specified in their laboratory Quality Assurance Plan. The manual discusses the schedule, procedures, criteria, and documentation in place at the laboratory to prevent instrument and equipment failure and to minimize downtime. For each instrument or piece of equipment, the laboratory maintains:

- instrument/equipment inventory list;
- list or inventory of major spare parts;



- external vendor service agreements (if applicable); and
- instrument-specific preventive maintenance logbook or file.

The laboratory documents all preventive maintenance of equipment in dedicated logbooks or files.

3.7 Instrument Calibration and Frequency

General guidance regarding calibration and frequency of calibration of both field and laboratory equipment are described in the following subsections.

3.7.1 Field Instruments

The field equipment that will need calibration for the perimeter air sampling program will include a PQ-100/200 air sampler. Proper maintenance, calibration, and operation of the instrument will be the responsibility of field sampling team personnel assigned to a particular field activity. Other equipment, such as a photoionization detector, dust monitor, landfill gas meter (for oxygen, carbon dioxide, methane, etc.), will be used as part of the project. All instruments and equipment used during this Project will be maintained, calibrated, and operated according to the manufacturer's guidelines and recommendations. Field equipment requiring regular calibration will be calibrated at least once per day. Relevant manuals will be kept with field sampling team personnel during the performance of field activities. Equipment will receive routine maintenance checks to minimize equipment breakdown in the field. Any items found to be inoperable will be taken out of use and a note stating the time and date of this action will be made in the daily field records. An equipment calibration daily log form for selected equipment is provided in Appendix D.

3.7.2 Laboratory Equipment and Instrumentation

All laboratory equipment and instruments specific to each analysis are included in methodspecific SOPs, which can be obtained from the laboratory directly.

Whenever possible, the laboratory uses recognized procedures for calibration, such as those published by U.S. EPA or the American Society of Testing Materials. If established procedures are not available, the laboratory develops a calibration procedure based on the type of equipment, stability, characteristics of the equipment, required accuracy, and the effect of operation error on the quantities measured. Whenever possible, the laboratory uses physical reference standards associated with periodic calibrations such as weights or certified



thermometers with known relationships to nationally recognized standards. When national reference standards are unavailable, the basis for the reference standard is documented.

Equipment or instruments that fail calibration or become inoperable during use are tagged to indicate they are out of calibration. Such instruments or equipment are repaired and successfully recalibrated prior to re-use.

3.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

Supplies and consumables that may be used during field investigations include sample bottles, calibration gases, hoses, materials for decontamination activities, DI water, and potable water. Project team members obtaining supplies and consumables are responsible for confirming that the materials meet the required specifications, are intact and in good condition, are available in adequate supply, and are stored appropriately until use. Project team members will direct any questions or any identified problems regarding supplies and consumables to the Task Leader for resolution.

3.9 DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

Non-direct measurements typically refer to non-direct data, such as historical investigation reports, historical maps, and site plans. Non-direct measured data is not anticipated for this work.

3.10 DATA MANAGEMENT

The objective of data management is to establish procedures to be used during field investigations for documenting, tracking, and presenting investigative data. Data generated during the field investigations, as well as previously existing data, will form the basis for developing conclusions and recommendations. Efficient utilization and comprehensive consideration of available data requires that the data be properly organized for review. Organization of the data shall be planned prior to collection to assure the generation of identifiable and useable data. This section describes procedures necessary to provide for collecting sufficient data to accurately validate raw data and to transfer validated data to a data management system through which it can be evaluated with minimal effort. This section also describes the operating practices to be followed by personnel while collecting and reporting data.



The flow of data for the Project will be as follows.

- Field notes will be forwarded to the Geomatrix PM.
- Soil, soil vapor, concrete, and air samples will be sent directly from the field to the selected laboratory. Copies of chain of custody forms and other field datasheets will be forwarded to Geomatrix.
- Laboratory results, including EDDs and hard copies, will be sent to the Geomatrix PM and Task Manager.
- Geomatrix will perform data verification, complete the data verification checklists (included in Appendix D), and indicate any resulting data qualifiers in the project database and on hard copies of laboratory reports.

3.10.1 Data Recording

Observations made and measurements taken in the field are recorded on appropriate activity-specific data sheets or in the field notes.

Data used for analysis, presentation, and reporting will be stored in an electronic database. This database will facilitate:

- reviewing and evaluating analytical data against project-specific criteria; and
- producing data tables and figures.

Laboratory results will be submitted as a complete and single EDD. It is expected that the laboratory will compare electronic data with the hard-copy report prior to submittal to confirm that the EDD and hard-copy data are identical. Geomatrix will check the EDD against the hard copy for all detected analytes. The EDD will be submitted on a diskette or via e-mail, with the disk label or email including the Laboratory Delivery Group, submittal date, laboratory name, and site description. If an EDD is resubmitted to Geomatrix, the EDD will be labeled "Revised."

3.10.2 Data Verification

Data verification, which is an integral part of the QA program, consists of reviewing and assessing the quality of data. Data verification provides assurance that the data as reported are of acceptable quality. For validity, the characteristics of importance are precision, accuracy, representativeness, comparability, and completeness. Data usability describes whether a dataset is sufficiently complete and of sufficient quality to support a decision or action in terms of the specific DQOs.



Analytical data submitted by the laboratory in electronic form will be verified, and, if necessary, exception reports will be produced. Qualified results will be loaded into the database.

The data verification process includes:

- evaluating against criteria for blanks—laboratory and field blanks;
- evaluating against accuracy criteria—holding times, surrogates, laboratory control samples, and MS;
- evaluating against precision criteria—MS/MSD, and field and laboratory duplicates;
- confirming that data qualifiers are assigned appropriately; and
- uploading analytical data only to the electronic database.

3.10.3 Data Transformation

Transforming data by converting individual data point values into related values or symbols using conversion formulas or a system of replacement is not currently proposed for data evaluation for the project at this time. If data transformation is required at a later date, then conversion procedures will be described in detail in the associated technical report.

3.10.4 Data Transmittal

Analytical data are provided by the laboratory in both a hard-copy and EDD format. The electronic data are to be provided in a specified format that will be uploaded to intermediate files, and then reviewed for completeness and accuracy by the PM before being validated and then uploaded to the Project database.

3.10.5 Data Analysis

Data analysis (e.g., computation of summary statistics, standard errors, confidence intervals, etc.) is not currently proposed for data evaluation for this Project at this time. If data analysis is required at a later date, then the analysis procedures will be described in detail in the associated technical report.

3.10.6 Data Tracking

The QA Advisor is responsible for data management. The QA Advisor has the authority to enforce proper procedures as outlined in this QAPP and to implement corrective procedures to



provide for the accurate and timely flow and transfer of data. The QA Advisor and PM will review final data reports.

Data will be generated from environmental sampling and analysis, field analyses, and field readings. The individuals who generate data (geologists, engineers, samplers, and chemical analysts) will be responsible for accurate and complete documentation of required data, and for assuring that those data are provided to their supervisor in a timely manner.

The Task Leader will be responsible for the day-to-day monitoring of data collected in the field. He/she assures that data are collected in the format specified in the Soil, Soil Vapor, and Concrete Sampling and Analysis Plan, assigns sample designations, and routes data to the project files. At least one copy of all project documents will be retained by the Task Leader for project use during the work activity. Original documents will be maintained in the project file.

The Task Leader will be responsible for the day-to-day monitoring of activities related to the generation and reporting of chemical data. He/she ensures that samples are analyzed according to the specified procedures; that data are verified; and that the data are properly coded, checked for accuracy, and entered into the data management system. He/she assures the data are then routed to the project files.

3.10.7 Data Storage and Retrieval

A project file will be established for storing original data, historical data, written documents, and data collected or generated during this work. Geomatrix maintains a central filing system in which the project file will be located.

All materials will be dated and will bear the project number. All documents relating to the project shall be controlled to provide proper distribution, filing, and retrieval. Document control shall also assure that revisions are properly recorded, distributed, and filed. The PM maintains overall responsibility for the project files and provides that appropriate documents are filed. Project files will be retained for a minimum of 5 years after the completion of the project.

4.0 ASSESSMENT AND OVERSIGHT

Internal and external checks (assessments) that have been built into this project to assure that:

 elements of this QAPP have been properly implemented as prescribed for all investigations;



- the quality of the data generated is adequate and satisfies the DQOs that have been identified in this QAPP; and
- corrective actions, when needed, are implemented in a timely manner and their effectiveness is confirmed.

Assessment activities may include surveillance, inspection, peer review, review of management systems, readiness review, technical systems audit, performance evaluation, and data quality assessment.

4.1 ASSESSMENT ACTIVITIES

The following subsections identify the assessment and oversight activities planned to provide that the objectives identified above are attained by field and laboratory operations. The QA Advisor, PM, and/or PIC may identify additional assessment activities to be performed during the project based on findings of the planned activities described below.

4.1.1 Assessment of Field Operations

In general, the QA Advisor and/or other designated members of the project team as appropriate will conduct internal assessments of field operations. The assessment activities for field operations will evaluate the following performance issues.

- Are sampling operations being conducted in accordance with the associated QAPP?
- Are the sample labels being filled out completely and accurately?
- Are the chain of custody records complete and accurate?
- Are the field memoranda being filled out completely and accurately?
- Are the sampling activities being conducted in accordance with the approved work plan?

Planned assessment activities to evaluate these and other field operations issues include surveillance (frequent review) of sample collection documentation, sample handling records (chain of custody forms), field notes, and field measurements, and the performance of unannounced audits of field operations.

The team member who conducts an assessment activity will report the results to the Task Leader and PM. Reports of assessment activities will include the findings and identification of any corrective actions taken or planned.



4.1.2 Assessment of Laboratory Operations

The laboratory has an ongoing internal audit programs implemented to monitor the degree of adherence to their own policies, procedures, and standards. The internal audit programs, described in the laboratory's Quality Assurance Plan, includes systems audits, performance evaluations, data audits, and spot assessments. Laboratory personnel who are independent of the area(s) being evaluated conduct internal audits. The laboratory also participates in external audits conducted by regulatory agencies and other clients. Project-specific assessments of laboratory operations are described below.

The Task Leader will be in frequent contact with the analytical laboratory during the time that samples are being analyzed. This regular contact will enable assessment of progress in meeting DQOs and early identification of any problems requiring corrective actions. The Task Leader will report promptly to the PM any identified problems, corrective actions taken, and recommendations for additional corrective actions. The PM will review the problem and provide for swift implementation of any outstanding corrective actions. The PM or Task Leader will be responsible for working directly with the laboratory to assure the prompt resolution of any problems identified.

4.2 REPORTS TO MANAGEMENT

This subsection discusses internal reports within the project team. External reports are discussed in Section 2.4.6.

Reports to management will include project status reports, the results of surveillance evaluations, field and/or laboratory audits, and data quality assessments. These reports will be directed to the PIC, who has ultimate responsibility for assuring that any corrective action response is completed, verified, and documented.

Final reports will include a QA section that describes:

- any problems that required corrective action and the resolution of those problems;
- an assessment of data quality in terms of precision and accuracy and how they affect the usability of analytical results;
- limitations on any qualified results and a discussion of any rejected results; and
- discussion of results of field and laboratory QA/QC samples.



5.0 DATA VALIDATION AND USABILITY

This section of the QAPP provides a description of the QA activities that will occur after the data collection phase of the project is completed. Implementation of this section is the responsibility of the QA Advisor and will determine whether or not the data conform to the specified criteria, thus satisfying the project objectives.

5.1 DATA REVIEW AND VERIFICATION

Data verification involves reviewing and accepting, qualifying, or rejecting data on the basis of sound criteria and following EPA guidelines. The laboratory will report data in data packages as described in Section 2.7.2. All of these data will be subject to limited data verification performed according to U.S. EPA Region 9 data evaluation and validation guidance, as discussed below.

Data verification will consist of a systematic review of the analytical results and associated QC methods and results. In any area not specifically addressed by EPA guidelines, best professional judgment will be utilized and described in the Usability Assessment portion of the data verification report.

Data verification will be conducted in accordance with the guidelines set forth with the latest version of the U.S. EPA Region 9 Data Evaluation/Validation Guidance (currently U.S. EPA Region 9, 2002a) and in compliance with the U.S. EPA National Functional Guidelines (U.S. EPA, 1999, 2002b). In general, data verifications will include a check of data completeness for each data package, a transcription check for sample results, and a thorough review of all laboratory reporting forms. Specifically, this review will include:

- review of data package completeness;
- review of required reporting summary forms to determine whether the QC requirements were met and to determine the effect of QC requirements on the precision, accuracy, and/or sensitivity of the data;
- review of the overall data package to determine whether contractual requirements were met;
- review of additional QA/QC parameters, such as blanks to assess the technical usability of the data; and
- application of standard data quality qualifiers to the data.



In addition, each data verification effort will include a comprehensive review of the following data quality indicators:

- holding times (to assess potential for degradation that could affect accuracy);
- blanks (to assess contamination of all compounds);
- system monitoring compounds (to assess method accuracy);
- laboratory-fortified blanks (to assess accuracy of a method and precision of the method relative to the specific sample matrix);
- compound RLs (to assess sensitivity compared to project-specific requirements);
 and
- field duplicate RPDs (to assess precision of the method relative to field sampling techniques, the specific sample matrix, and representativeness of the sample aliquot to the area sampled).

The results of the data verification and any corrective actions implemented will be recorded on a QA/QC worksheet, which will be initialed and dated by the data reviewer. The QA Advisor or appropriate designee will provide secondary review of the QA/QC worksheet and will also initial and date the worksheet. The initialed and dated QA/QC worksheet will be attached to the final analytical laboratory report that is retained in the project files.

5.2 VERIFICATION METHODS

Data verification is conducted to assess the effect of the overall sampling and analysis process on the usability of the data. There are two areas of review: laboratory performance and the effect of matrix interferences. Evaluation of laboratory performance is a straightforward examination for compliance with the method requirements. The laboratory either did or did not analyze the samples within the QC limits of the analytical method and according to protocol requirements. The assessment of potential matrix effects consists of a QC evaluation of the analytical results and the results of blank, duplicate, and matrix spike samples. Data verification is at times based on best professional judgment. To provide consistent data verification, worksheets will be completed for each data verification effort. A data review worksheet is a summary form on which the data reviewer records notes and conclusions specific to each analytical method. The worksheets will help the reviewer track and summarize the overall quality of data. Sample results will then be qualified as appropriate, following EPA protocols. Samples that do not meet the acceptance limit criteria will be annotated with a



qualifying flag, which is a one- or two-letter abbreviation that indicates a problem with the data (Table 4).

During verification, the entire dataset will be examined for overall trends in data quality and usability. Information summarized as part of the data quality verification will include frequencies of detection, dilution factors that might affect data usability, and patterns of target compound distribution. The dataset also will be evaluated to identify potential data limitations or uncertainties in the laboratory procedures.

All analytical data will be supported by a data package. The data package will contain the supporting QC data for the associated field samples. Data verification will be documented with:

- a completed data review worksheet;
- a comprehensive narrative detailing all QC exceedances and explaining qualifications of data results. In cases where data are qualified because of quantifiable QC exceedances, the bias (high or low) will be identified;
- data summaries in tabular format reporting all data results with the qualifiers that
 were added during data review. These tables will include sample ID, laboratory ID,
 date sampled, sample type (e.g., field duplicate, field blank), units, concentration of
 analytes, and qualifiers. The tables may be modified to report other appropriate
 information (such as depth of discrete-depth samples, date analyzed, dilution
 factor); and
- requests to resubmit sent to the laboratory for missing information, verification of analytical information, etc.

5.3 RECONCILIATION WITH USER REQUIREMENTS

The usability of the verified data will be assessed by comparing the data to the verification criteria and DQOs. The usability assessment will provide an overall summary of data quality, defining acceptability or problems with accuracy, precision, sensitivity, and/or representativeness of the results and providing clear guidance to the data users on any uncertainties in data that have been qualified as estimated. Because of the cumulative effects of QC exceedances, some specific results may be determined to be unusable. Alternatively, based on EPA guidelines and best professional judgment, specific results may be determined to be usable for DQOs when they are not significantly outside the QC criteria.



The final step of the data verification process is to assess whether the data meet the DQOs. The final results, adjusted for the findings of data verification, will be compared to the DQOs to assess whether the data are of sufficient quality to support the DQOs. The decision regarding data sufficiency may be affected by the overall precision, accuracy, and completeness of the data as demonstrated by the data verification process. If the data are sufficient to achieve project objectives, the PM will release the data and work can proceed. If the data are insufficient, corrective action will be required.



6.0 REFERENCES

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SAMPLE ANALYTICAL METHOD INFORMATION

Former Pechiney Cast Plate, Inc. Vernon, California

Target Analytes	Media	Analytical Method	Sample Volume; Container/Preservation	Reporting Limits	Holding Time
TPH¹ with carbon chain range quantification (TPHcc)	Soil	EPA ² 8015M	4 oz ³ glass jar; unpreserved	Listed in Laboratory Quality Assurance Manual	14 days
TPH as Stoddard Solvent	Soil	EPA 8260M/5035	4 oz glass jar; unpreserved	Listed in Laboratory Quality Assurance Manual	14 days
Volatile Organic Compounds	Soil	EPA 8260B/5035	4 oz glass jar; unpreserved	Listed in Laboratory Quality Assurance Manual	14 days
Volatile Organic Compounds ⁴	Vapor	EPA 8260 (Modified)	125 milliliters glass bulb or 100 cc sampling syringe	Listed in Laboratory Standard Operating Procedures (Appendix B)	15 minutes or 4 hours (with surrogates)
PCBs⁵	Soil	EPA 8082	4 oz glass jar; unpreserved	Listed in Laboratory	40 days (extraction within 14 days)
PCBs	Concrete	EPA 8082	0.8 to 1.2-inch diameter 3-inch depth	Quality Assurance Manual	40 days (extraction within 14 days)
CAM Metals ⁶	Soil	EPA 600B/7000 ⁷	4 oz glass jar; unpreserved	Listed in Laboratory Quality Assurance Manual	180 days
Semi-volatile Organic Compounds	Soil	EPA 8270C	4 oz glass jar; unpreserved	Listed in Laboratory Quality Assurance Manual	40 days (extraction within 14 days)
PM-10 ⁸ Particulates	Dust	NIOSH ⁹ 0500	47 mm ¹⁰ diameter Teflon filter; unpreserved	6 μg/m ^{3 11}	180 days
Lead	Air	NIOSH 7300	47 mm diameter Teflon filter; unpreserved	$0.1 \mu\text{g/m}^3$	180 days
PCBs	Air	TO-10A	60 mm length x 20 mm diameter glass tube; unpreserved	0.6 μg/m ³	40 days (extraction within 7 days)
Volatile Organic Compounds – TCE, PCE, Benzene, 1,2,4- TMB, 1,3,5-TMB	Air	TO-15	6 L summa canister; unpreserved	$\begin{aligned} & PCE^{12} - 0.27 \ \mu g/l^{13} \\ & TCE^{14} - 0.5 \ \mu g/l \\ & Benzene - 0.029 \ \mu g/l \\ & 1,2,4 - TMB^{15} - 0.062 \ \mu g/l \\ & 1,3,5 - TMB^{16} - 0.062 \ \mu g/l \end{aligned}$	30 days

Notes:

- 1. TPH = Total Petroleum Hydrocarbons.
- 2. EPA= Environmental Protection Agency.
- 3. oz = ounce.
- 4. Regional Water Quality Control Board target list of 23 VOCs (Phase I and IV Areas) and Stoddard solvent and associated VOCs (Phase IV Area) using gas chromatography/mass spectrometry methods similar to EPA Method 8260B.
- $5. \ \ PCBs = Polychlorinated \ Biphenyls.$
- 6. CAM Metals = Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Cobalt, Copper, Lead, Molybdenum, Nickel, Selenium, Silver, Thallium, Tin, Vanadium, and Zinc.
- 7. Mercury Total analyzed by EPA 7470A/7471A.
- 8. PM-10 = particulate matter less than 10 microns.
- 9. NIOSH = National Institute for Occupational Safety and Health.
- $10. \ mm = millimeter.$
- 11. $\mu g/m3 = micrograms per cubic meter.$
- 12. PCE = tetrachloroethylene.
- 13. $\mu g/l = micro grams per liter.$
- 14. TCE = trichloroethylene.
- 15. 1,2,4 TMB = 1,2,4 trimethylbenzene.
- 16. 1,3,5 TMB = 1,3,5 trimethylbenzene.



METHOD PERFORMANCE OBJECTIVES ACCEPTANCE CRITERIA

Former Pechiney Cast Plate, Inc. Vernon, California

Method Performance Objective	Type of Quality Control Sample	Frequency	Acceptance Criteria
Precision—Field	Duplicate field sample	1 per 10 samples (soil vapor only)	Relative percent difference (RPD) <30
Precision—Laboratory	Laboratory control samples (LCS) and laboratory control duplicate (LCSD) samples	1 per batch of 20 samples per matrix	RPD <30
	Matrix spike (MS) and matrix spike duplicate (MSD) samples	1 per batch of 20 or fewer investigative samples per matrix	RPD <30
	Unspiked duplicate samples	1 per batch of 20 samples per matrix	RPD <30
Accuracy—Field	Trip blanks	1 per cooler of volatile organic compounds (VOC) samples	U.S. Environmental Protection Agency (U.S. EPA) National Functional Guidelines Protocol
	Equipment rinsate blank	1 per day per equipment type (excluding excavation equipment)	U.S. EPA National Functional Guidelines Protocol
	Field decontamination water blank	1 per water source per sampling event (per the discretion of the Project Manager)	U.S. EPA National Functional Guidelines Protocol
Accuracy—Laboratory	Matrix spike (MS) samples	1 per batch of 20 or fewer investigative samples per matrix	Percent recovery (%R) less than compound specific limit (See Laboratory Quality Assurance Manual)
	Laboratory control samples (LCS)	at least once with each analytical batch, with a minimum of 1 for every 20 samples	%R less than compound specific limit (See Laboratory Quality Assurance Manual)
	Method blanks	at least once with each analytical batch, with a minimum of 1 for every 20 samples	No compounds should be detected in laboratory method blanks
	Preparation blanks	at least once with each analytical batch, with a minimum of 1 for every 20 samples	%R less than compound specific limit (See Laboratory Quality Assurance Manual)
	Surrogates		%R less than compound specific limit (See Laboratory Quality Assurance Manual)
Representativeness	Not applicable	Not applicable	Numerical goals cannot be used to evaluate this subjective measure.
Completeness	Not applicable	Not applicable	90% completeness
Comparability	Not applicable	Not applicable	Comparable if the same procedures for collecting and analyzing the samples are used, if the samples comply with the same QA/QC procedures, and if the units of measurement are the same
Sensitivity	Not applicable	Not applicable	Reporting limits (RLs) below or equal to the task-specific target analysis goals or concentrations



FIELD QUALITY CONTROL SAMPLES

Former Pechiney Cast Plate, Inc. Vernon, California

Soil Samples

Type of Quality Control	Frequency	Acceptance Criteria
Sample		
Trip blanks	1 per cooler of VOC samples	U.S. EPA National Functional Guidelines
		Protocol
Equipment rinsate blank	1 per day per equipment type that is	U.S. EPA National Functional Guidelines
	decontaminated	Protocol
Field decontamination	1 per water source per sampling event	U.S. EPA National Functional Guidelines
water blank	(per the discretion of the Project	Protocol
	Manager)	
Temperature Blank	1 per day per cooler	U.S. EPA National Functional Guidelines
		Protocol

Air Samples

Type of Quality Control Sample	Frequency	Acceptance Criteria
Temperature Blank	1 per day per cooler with PCB samples	U.S. EPA National Functional Guidelines
		Protocol
Field blank	1 per month	U.S. EPA National Functional Guidelines
		Protocol

Notes:

PCB = polychlorinated biphenyl VOCs = volatile organic compounds

U.S. EPA = U.S. Environmental Protection Agency



DATA QUALIFIER DEFINITIONS

Former Pechiney Cast Plate, Inc. Vernon, California

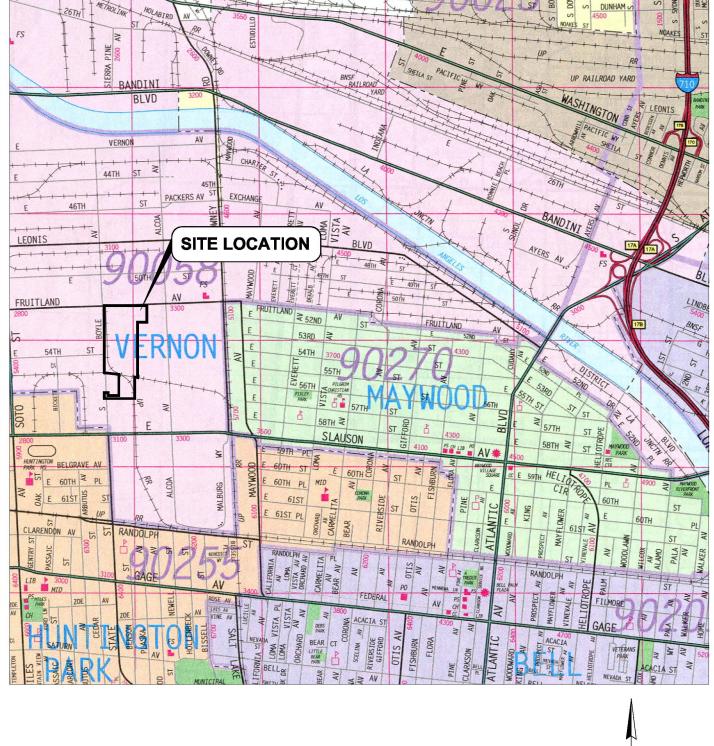
Qualifier	Explanation of Qualifier
Organic Analyses 1	
U	The compound was analyzed for, but was not detected above the reported sample quantitation limit.
Ј	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
Inorganic Analyses ²	
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
Ј	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
ΠΊ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.

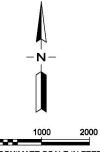
Notes

- 1. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, USEPA 540-R-99-008, October 1999.
- 2. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, USEPA 540-R-01-008, July 2002.



FIGURES





APPROXIMATE SCALE IN FEET

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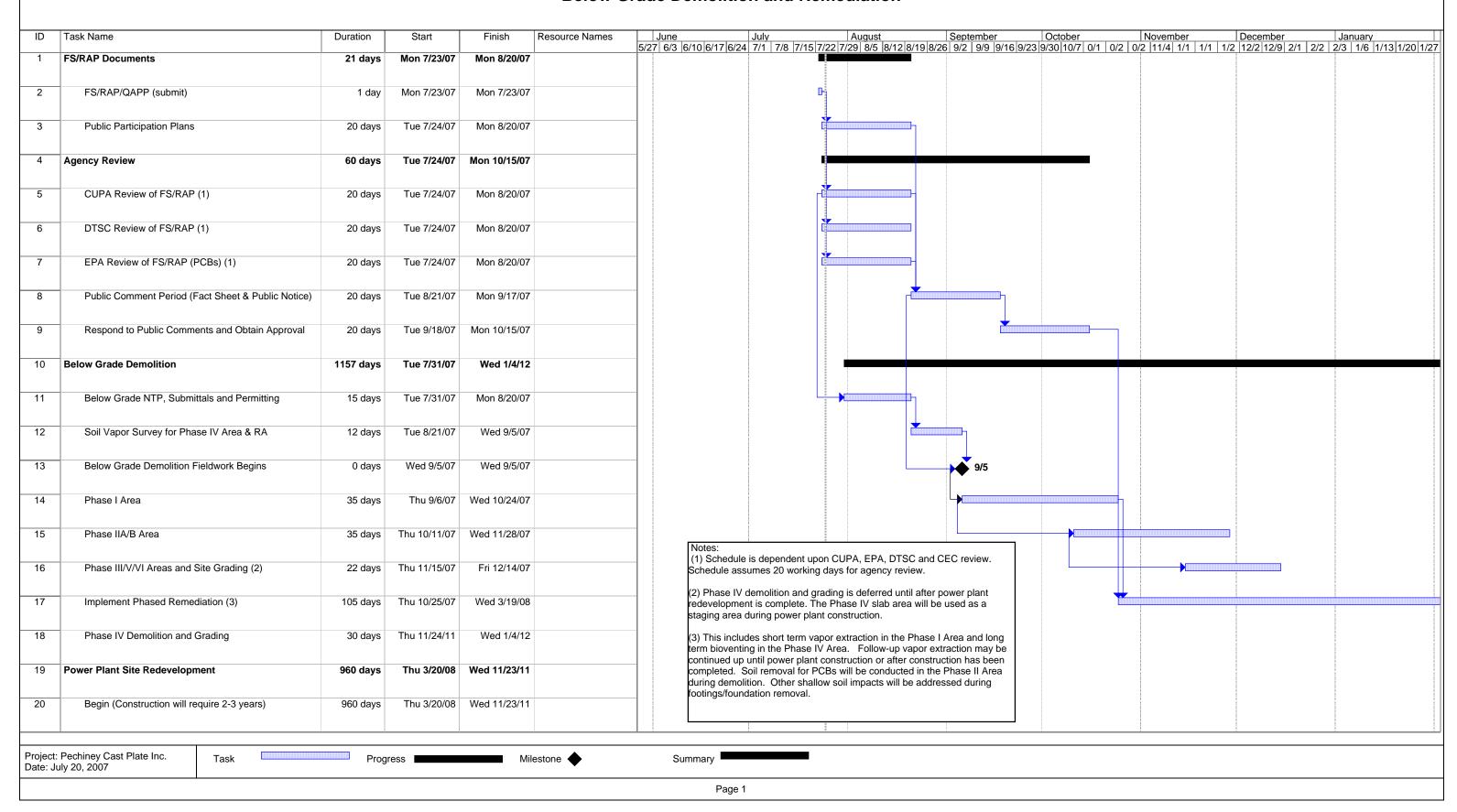
SITE LOCATION MAP

PECHINEY CAST PLATE Vernon Facility, 3200 Fruitland Avenue Vernon, California

DI PERIMISSION	•
Figure By jrW	Project No. 10627
Date 8/31/05	Figure 1

Geomatrix

Figure 3 Proposed Schedule Former Pechiney Cast Plate, Inc. Facility Below Grade Demolition and Remediation





APPENDIX A

LABORATORY QUALITY ASSURANCE MANUALS



AMERICAN ANALYTICS

QUALITY ASSURANCE/ QUALITY CONTROL MANUAL

APRIL 1993

(Revision 2, December 19, 1996) (Revision 3, December 18, 1998) (Revision 4, February 9, 2000) (Revision 5, September 19, 2000) (Revision 6, December 12, 2001) (Revision 7, July 22, 2002) (Revision 8, October 4, 2002)

(Revision 9, March 25, 2003)

(Revision 10, December 14, 2004)

MANQAQC01 REV 10

Written by:	Ml.		Approved t		Allo	n.A
willon by.	Lab Director		Approved k	QA/QC	Officer	
Copy No.:	01	Submitted to:	Geomatrix	Consult.	Date: _	2/15/07

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1.0 PROGRAM DESCRIPTION

American Analytics provides laboratory services for environmental analysis. The laboratory occupies approximately 6500 square feet of a facility at 9765 Eton Avenue, Chatsworth, CA 91311. American Analytics performs environmental analytical tests in various sample matrices including: soils, sludges, water, wastewater, drinking water, vapors and hazardous materials. The laboratory performs the analyses in accordance with methods specified in the EPA manual, Methods for Analyzing Hazardous Waste (SW-846, Third Edition, 1986 and SW-846, Update III, Revision 1, December 1996), the EPA methods published in the Federal Register (CFR 40 Part 136, October 26, 1984), the Standard Methods for the Examination of Water and Waste Water (American Public Health Association) and other official public testing procedures. The analyses performed by American Analytics are listed in Tables 1, 2, and 3.

Table 1
Organic Analyses Performed by American Analytics

EPA Method	Compound Class
8010, 601	Halogenated Volatile Organics
8015	Fuels and Nonhalogenated Volatile Organics
8020, 602	Aromatic Volatile Organics
8021B	Halogenated and Aromatic Volatile Organics
8081A	Organochlorine Pesticides by GC
8082	Polychlorinated Biphenyls (PCB's) by GC
8240, 8260B, 624	Volatile Organics by GC/MS
8270C, 625	Semi Volatile Organics by GC/MS
8310, 610	Polynuclear Aromatic Hydrocarbons (PAHs) by HPLC
524.2	Volatile Organic Compounds in Drinking water
CDHS SRL PT/GCMS	1,2,3-Trichloropropane
8270M	1,4-Dioxane by Isotope Dilution
1625M	NDMA by GCMSMS

Table 2
Inorganic Elemental Analyses Performed by American Analytics

Parameter	EPA Method
Aluminum	202.1, 202.2, 7020, 6010, 200.7
Antimony	204.1, 204.2, 7040, 6010, 200.7
Arsenic	206.2, 206.3, 7060, 6010, 200.7
Barium	208.1, 208.2, 7080, 6010, 200.7
Beryllium	210.1, 210.2, 7090, 6010, 200.7
Boron	200.7
Cadmium	213.1, 213.2, 7130, 6010, 200.7
Calcium	215.1, 200.7
Chromium	218.1, 218.2, 7190, 6010, 200.7
Chromium (VI)	218.4, 218.5, 218.6, 7196, 7199
Cobalt	219.1, 219.2, 7200, 6010, 200.7
Copper	220.1, 220.2, 7210, 6010, 200.7
Iron	236.1, 236.2, 7380, 6010, 200.7
Lead	239.1, 239.2, 7420, 7421, 6010, 200.7
Lithium	6010
Magnesium	242.1, 200.7
Manganese	243.1, 243.2, 200.7
Mercury	245.1, 245.2, 245.5, 7470, 7471
Molybdenum	246.1, 246.2, 7480, 6010, 200.7
Nickel	249.1, 249.2, 7520, 6010, 200.7
Phosphorous	6010
Potassium	258.1, 200.7
Selenium	270.2, 270.3, 7740, 6010, 200.7
Silver	272.1, 272.2, 7760, 6010, 200.7
Silica	200.7
Sodium	273.1, 200.7
Strontium	6010
Thallium	279.1, 279.2, 7840, 6010, 200.7
Tin	282.1, 282.2, 7870
Vanadium	286.1, 286.2, 7910, 6010, 200.7
Zinc	289.1, 289.2, 7950, 6010, 200.7

Table 3
General Chemistry Analyses Performed by American Analytics

Parameter	EPA Method	
California Waste Extraction Test (WET)	Title 22	
Toxicity Characteristic Leaching Procedure (TCLP)	1310, 1311	
Alkalinity	SM 2320 B	
Ammonia	350.3	
BOD	405.1, SM 5210B	
Chloride	325.3/300.0	
Chlorine Residual	330.2/330.3	
COD	410.4, SM 5220	
Cyanide	335.1, 335.2, SM 4500	
Fluoride	340.2	
Hardness – Total as CaCO3	130.2/200.7	
Nitrate	353.3/300.0	
Nitrite	354.1/300.0	
Perchlorate	314.0	
Oil & Grease	413.1/413.2	
Oxygen Dissolved	360.1	
рН	150.1, 9040, 9041, 9045	
Phosphate – Ortho	300.0	
Residue, Total (TS)	160.3	
Residue, Filterable (TDS)	160.1	
Residue, Nonfilterable (TSS)	160.2	
Residue, Settleable (SS)	160.5	
Specific Conductance	120.1	
Sulfate	375.4/300.0	
Sulfide	SM 4500/376.2	
Ferrous Iron	SM 3500	
Turbidity	180.1	
Total Recoverable Petroleum Hydrocarbons (TRPH)	418.1	

American Analytics is committed to producing the highest quality analytical product possible. It is important that our product meets and surpasses the analytical needs of our clients, and that our data is reliable and legally defensible. In addition, American Analytics is committed to continually improving its work product by using the latest analytical technology, keeping up to date on current environmental regulations, and improving our client service.

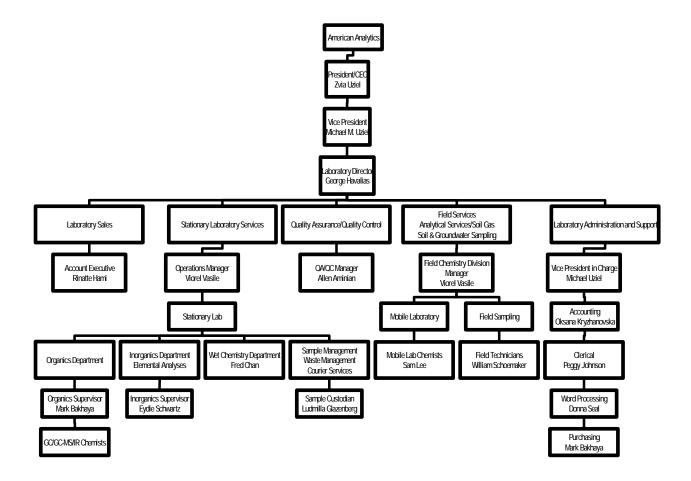
2.0 PROGRAM ORGANIZATION AND RESPONSIBILITY

The vice president and laboratory director of American Analytics are responsible for major decisions concerning the laboratory. The overall performance of the laboratory is the responsibility of the operations manager who monitors the day-to-day operations of the laboratory.

The QA/QC officer under the supervision of the laboratory director is responsible for the implementation and maintenance of the Quality Assurance/Quality Control (QA/QC) program. The QA/QC officer is also responsible for reviewing and updating the QA/QC manual as necessary when changes are made to existing QA/QC practices in the laboratory. This is necessary since the American Analytics QA/QC program is dynamic and changing as necessary to continuously improve the quality of work in the laboratory.

The organics department supervisor is responsible for the validation of all data generated in the organics section of the laboratory. The inorganics department supervisor is responsible for the validation of all data generated in the inorganics section of the laboratory. The analytical chemists are trained in the Standard Operating Procedures (SOPs) and supervised by the operations manager and the section supervisors. All personnel are experienced in trace analytical chemistry and environmental analysis.

Figure 1
American Analytics Organization Chart



3.0 PERSONNEL AND QUALIFICATIONS

3.1 The chief laboratory personnel are listed below:

Michael M. Uziel, Ph.D., Vice President

Dr. Uziel, Vice President of American Analytics, is a graduate of the University of California, Berkeley with a Ph.D. and M.S. in civil engineering, and a B.S. in biochemistry. He has over 20 years experience in the toxic waste industry and analytical chemistry.

Dr. Uziel has worked in both Southern and Northern California investigating underground tank leaks, performing soil and groundwater investigations and cleanups, soil gas surveys, developing and implementing remedial investigation/ feasibility studies (RI/FS). He has a great deal of experience working with such agencies as the State Regional Water Quality Control Board and the State Department of Health Services. He also worked on planning chemical analysis work including QA/QC programs on several federal Superfund toxic waste sites.

Dr. Uziel's experience in analytical chemistry goes beyond his recent experience. In the early seventies, Dr. Uziel worked as an analytical chemist at the Department of Hydraulics and Sanitary Engineering at the University of California, Berkeley. As part of his work, Dr. Uziel planned and constructed the analytical chemistry laboratory of the department. Later he served as a teaching assistant in undergraduate and graduate courses in analytical chemistry at the university. Dr. Uziel's doctoral research included extensive personal use of gas chromatography systems, and other analytical techniques at U.C. Berkeley - Richmond field station. As an honor student in Biochemistry (U.C. Berkeley), Dr. Uziel performed biochemical research where he extensively used analytical equipment in the biochemistry department and biodynamics laboratory of U.C. Berkeley.

George Havalias, Laboratory Director

Mr. Havalias is a graduate of the University of Missouri at Rolla with a B.S. in chemical engineering. He has over twelve years experience in the area of environmental analyses at both state and commercial laboratory levels. He has also served as a process/project engineer in the field of petrochemical refinery design for a major engineering firm in Southern California.

As laboratory director, Mr. Havalias oversees the QA/QC program at American Analytics and is responsible for monitoring and improving the quality of the analytical data produced by the laboratory. He also assists with the training of new personnel in existing methods, and is actively involved with bringing new methods on-line to broaden the spectrum of analytical testing performed by the laboratory.

Allen Aminian, QA/QC Officer, Technical Director

Mr Aminian has a Bachelor of Science degree from the University of Kansas. He has fifteen years of experience in the environmental industry with emphasis on organic analyses, including EPA methods 8260, 8270, 8081 and 8015. Allen is well versed in all aspects of the laboratory operations and is responsible for the implementation, maintenance, and continuing improvement of the quality assurance/quality control program at American Analytics. This process is accomplished by working in coordination with the laboratory director. Specific responsibilities include: updates to the QA/QC manual, writing and updating standard operating procedures, internal quarterly audits, establishing control limits and charts, coordinate and oversee the analysis and reporting of check samples.

Viorel Vasile, Operations Manager, Field Chemistry Division Manager

Mr Vasile has a Masters degree in Chemical Engineering from the University of Bucharest. Viorel coordinates and oversees the day to day laboratory operations and is responsible for the quality and on time delivery of analytical results to the client. Viorel also coordinates all aspects of field mobile laboratory operations including: Scheduling of field operations, client interface, vehicle maintenance, instrument maintenance, chemist training and quality assurance/quality control elements of field chemistry operations.

Eydie Schwartz, Inorganics Section Supervisor

Ms. Schwartz is a graduate of the California State University at Long Beach, with a Masters degree in Microbiology. She has over fifteen years of experience in the field of environmental analytical chemistry and has worked at both the analytical chemist and supervisory levels. As supervisor, she is responsible for all aspects of the day to day operations of the Inorganics division of the laboratory. She is responsible for scheduling the work, data review and meeting turnaround time commitments for analytical results. She is also responsible for instrument maintenance, method development, and training of new personnel.

3.2 Personnel Responsibilities

<u>Laboratory Director</u> - supervise and manage laboratory operations, laboratory work quality, client interface, final report approval and signature.

<u>Operations Manager</u> – Oversee laboratory workflow process, manage day - to - day laboratory operations for all sections of the laboratory, meet client specific project requirements. Assemble and review final reports ensuring that all quality standards are met.

QA/QC Officer – Implement and maintain the QA/QC program in the laboratory.

<u>Section Supervisor</u> - supervise and manage day - to - day operations of respective laboratory department, second party review of laboratory analytical data.

<u>Chemist</u> - perform analyses, review raw data, data reduction, and reporting to electronic LIMS system.

<u>Sample Custodian</u> - receive all laboratory samples, check samples and chains-of-custody, sample log in and storage, project number initiation.

<u>Waste Management Coordinator</u> - organize and categorize laboratory wastes, control waste storage, manage waste pick-up and disposal.

3.3 Personnel Training

All laboratory personnel are trained directly by the Sections Supervisor or the Operations Manager on laboratory safety, analytical techniques, use of analytical equipment, data compilation, data reduction, LIMS system operation, and all other laboratory operations. Certain laboratory personnel are required to take appropriate 40 hour or 24 hour OSHA training courses depending on their responsibilities. Refresher courses are taken every year. Documentation on this OSHA training is maintained in each employee's personnel file.

New, experienced laboratory personnel are required to demonstrate their capabilities to the Operations Manager and/or Laboratory Director prior to beginning any work on their own. New, inexperienced laboratory personnel are also required to work with the Operations Manager, Laboratory Director, and/or other experienced chemists prior to working on their own. All new employees must work under direct supervision until they have demonstrated the ability to perform analyses or other work properly.

As previously mentioned, all training records are maintained in each employee's personal file. In addition, employee reviews and/or performance evaluations are documented in the personal files.

4.0 SAFETY

Safety in the laboratory is the primary consideration of American Analytics. The laboratory is furnished with a state-of-the-art environment, as well as the necessary protection against any accident. The staff is trained in the handling of hazardous materials, and emergency and response procedures, in the event of an accident.

For safety reasons, all the work involving chemicals is confined to designated areas equipped with chemical hoods and special air exchange ventilation.

All hazardous materials are disposed of per California Department of Health Services (CDOHS) regulations and transported to legal Class I disposal or recycling facilities. Manifest records are kept in the laboratory files.

5.0 QA/QC OBJECTIVES FOR MEASUREMENT DATA

The effectiveness of a QA/QC program is measured by the quality of the data generated by the laboratory. Data quality is judged in terms of precision, accuracy, representativeness, completeness, and comparability. These terms are defined and described as follows:

- **Precision:** Is the degree to which the measurement is reproducible. Actual control limits for the precision will depend upon the specific method; in general, the relative percent difference (RPD) should be within 20%, the limit set by the EPA for the Contract Laboratory Program (CLP).
- Accuracy: Is a determination of how close the measurement is to the true value. Unless specified otherwise in special contracts and particular methods, American Analytics parameter for accuracy is ± three standard deviations from the mean, with two standard deviations established as a warning for system check.
- Representativeness: Is the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the sample analyzed regardless of the heterogeneity of the original sample matrix. For example, with samples consisting of several phases, it may be advisable to analyze each phase separately and to determine each phase proportionately in terms of the whole sample.
- Comparability: Expresses the confidence with which one data set can be compared to another data set of the same property. Comparability is assured through the use of established and approved analytical methods, consistency in the basis of analysis (wet weight, volume, etc.), and consistency in reporting units (ppm, ppb, etc.).
- Completeness: Completeness is a measure of the percent of valid or usable data in relation to all information obtained for a valid scientific study. For completeness, it is expected that the methodology proposed for chemical characterization of the samples collected will provide data meeting QC acceptance criteria following standard laboratory data review and validation for at least 95% of all samples collected. Completeness may also be defined as a

comparison of the number of tests successfully completed (with acceptable QC) to the total number of tests requested.

• **Method Detection Limit:** Method detection limits are determined for all methods performed in the laboratory. For each method, a seven replicate study is performed by which the recovery of the parameters of interest are calculated and used to obtain the detection limits.

The quality objectives for the analyses conducted in the laboratory are presented in Table 4.

Table 4
Precision, Accuracy and Completeness Objectives

Reference	Analysis	Precision	Accuracy	Completeness
AA SOP#		RPD	(%LCS Recovery)	(% Val. Data)
MET01	ICP Metals (Al, Ba, Ca, Cr, Cu, Pb, Mn, Ni, Tl, Sb, Be, Cd, Co, Fe, Mg, Mo, K, Na, V, Zn)	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%
MET02	Flame Metals (Al, Be, Cr, Fe, Mn, K, V, Sb, Ca, Co, Pb, Mo, Na, Zn, Ba, Cd, Cu, Mg, Ni, Tl, Ag)	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%
MET03	Graphite Furnace Metals (As, Be, Cr, Cu, Se, Tl, Sb, Cd, Co, Pb, Ag)	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%
MET07	Cold Vapor Metals (Hg)	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%

Reference	Analysis	Precision	Accuracy	Completeness
AA SOP#		RPD	(%LCS Recovery)	(% Val. Data)
IC01	Anions by IC Nitrate Nitrite Sulfate Phosphate Chloride Bromide	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%
IC03	Hexavalent Chromium by IC	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%
IC02	Perchlorate By IC	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%
IC05	Divalent Manganese By IC	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%

Reference	Analysis	Precision	Accuracy	Completeness
AA SOP#		RPD	(%LCS Recovery)	(% Val. Data)
IC06	Volatile Fatty Acids by IC	25% Aqueous Samples; 40% Nonaqueous or historical control limits	80-120% or Historical Control Limits	95%
GEN14	Alkalinity	0=0/		2=0/
GEN08	Ammonia	25% Aqueous	80-120% or	95%
GEN12	BOD	Samples;	Historical Control	
GEN17	Ferrous Iron	40%	Limits	
GEN18	Chlorine Residual	Nonaqueous or historical		
GEN13	COD	control limits		
GEN21	Cyanide			
GEN07	Fluoride			
GEN15	Hardness – Total as CaCO3			
GEN22	Oil & Grease by IR			
GEN16	Oil & Grease Gravimetric			
GEN22	Total Rec. Petr. Hydrocarbons			
GEN20	Oxygen Dissolved			
GEN01	рH			
GEN03	Residue, Total (TS)	250/	90 4200/	95%
GEN03	Residue, Filterable (TDS)	25% Aqueous Samples;	80-120% or Historical Control	
GEN013	Residue, Nonfilterable (TSS)	40% Nonaqueous	Limits	
GEN05	Residue, Settleable (SS)	or historical control limits		

Reference	Analysis	Precision	Accuracy	Completeness
AA SOP#		RPD	(%LCS Recovery)	(% Val. Data)
GEN02	Specific Conductance			
GEN11	Sulfide			
GEN06	Turbidity			
GEN04	Suspended Solids	25%	80-120%	95%
GEN09	Nitrate by Cadmium Reduction	25%	80-120%	95%
GEN10	Nitrite colorometric	25%	80-120%	95%
GEN19	Hexavalent Chromium colorometric	25%	80-120%	95%
GC01 GC04	Volatile Organics by GC	30% Aqueous Samples; 50% Nonaqueous or historical control limits	Historical Control Limits Or Method Specific Limits	95%
HP01 HP02	Polynuclear Aromatic Hydrocarbons (PAHs) by HPLC	30% Aqueous Samples; 50% Nonaqueous or historical control limits	Historical Control Limits Or Method Specific Limits	95%
GC05 GC06	Semi-Volatile Organics (Organochlorine Pesticides and PCB's) by GC	30% Aqueous Samples; 50% Nonaqueous Or historical Control limits	Historical Control Limits Or Method Specific Limits	95%
GC08	Alcohols by GC/FID	30% Aqueous Samples; 50% Nonaqueous Or historical Control limits	Historical Control Limits Or Method Specific Limits	95%

Reference	Analysis	Precision	Accuracy	Completeness
AA SOP#		RPD	(%LCS Recovery)	(% Val. Data)
MS01 MS02	Volatile Organics by GC/MS	30% Aqueous Samples; 50% Nonaqueous or historical control limits	Historical Control Limits Or Method Specific Limits	95%
MS09	Volatile Organics in Drinking Water by GC/MS	30%	Historical Control Limits Or Method Specific Limits	95%
MS06	1,2,3- Trichloropropane by GCMSMS	30%	Historical Control Limits Or Method Specific Limits	95%
MS03 MS05	Semi-Volatile Organics by GC/MS	40% Aqueous Samples; 60% Nonaqueous or historical control limits	Historical Control Limits Or Method Specific Limits	95%
MS04	1,4-Dioxane by Isotope dilution	30%	Historical Control Limits Or Method Specific Limits	95%

6.0 SAMPLE CUSTODY AND HANDLING

In the laboratory and in the field, correct sampling, handling, and storage of the samples are essential to produce reliable concentration data for the samples. American Analytics sample collecting and sample handling procedures are as follows:

- Sample Collection
- Sample Preservation
- Sample Custody
- Sample Handling

6.1 Sample Collection

Sampling equipment, appropriate containers, appropriate preservatives, and careful monitoring of holding times are a few of the points which must be considered in order to minimize possible contamination or other threats to the integrity of the sample. Proper sample collection and handling is the responsibility of the sample collector, who must follow EPA guidelines. If the sample collector requires assistance, American Analytics will provide written instructions for sample collection, handling, and storage, as well as, proper sample containers. The guidelines for sampling are given in Chapter Four of EPA Manual SW-846, and are summarized in Appendix A. In addition, Chapter Four of SW-846 presents general information on sampling techniques and guidelines. Any client requiring assistance will be referred to these references, and/or provided copies of the same if unable to obtain copies of documents before analysis.

6.2 Sample Preservation

The guidelines followed for sample preservation can be found in Chapter Four of EPA Manual SW-846 and are summarized in Table 4. Preservation techniques are usually performed in the field when the samples are collected.

Table 5
Sample Handling Procedures

EPA Method	Sample Container ¹	Sample Preservation	Preferred Volume	EPA Holding Time ⁴
		Volatile Organics		
601/8010	VOA	Store @ 4°C (2)	3 x 40 ml	14 days
8015M (Gasoline	VOA	Store @ 4°C	3 x 40 ml	14 days

EPA Method	Sample Container ¹	Sample Preservation	Preferred Volume	EPA Holding Time ⁴
Range)				
602/8020	VOA	Store @ 4°C (2,3)	Store @ 4°C (2,3) 3 x 40 ml	
8021	VOA	Store @ 4°C (2,3)	3 x 40 ml	14 days
8240/8260/524.2	VOA	Store @ 4°C (2,3)	3 x 40 ml	14 days
624	VOA	Store @ 4°C (2,3)	3 x 40 ml	14 days
	Se	mi-Volatile Organics		
8081/8082	1L Amber	Store @ 4°C	1000 ml	7/40 days
625/8270/1625M	1L Amber	Store @ 4°C	1000 ml	7/40 days
8310/610	1L Amber	Store @ 4°C	1000 ml	7/40 days
8015M (Diesel Range)	1L Amber	Store @ 4°C	1000 ml	7/40 days
TRPH (418.1)	1L Amber	Store @ 4°C, H ₂ SO ₄	1000 ml	28 days
	(General Chemistry		
pН	1L Plastic/Glass	Store @ 4°C	100 ml	Immediately
Mercury	1L Plastic	HNO ₃ to pH<2	500 ml	28 days
Chromium (VI)	1L Plastic	Store @ 4°C	500 ml	24 hours
Organic Lead	1L Plastic	Store @ 4°C	1000 ml	14 days
All other metals	1L Plastic	HNO ₃ to pH<2	1000 ml	6 months
Alkalinity	250 ml Plastic	Store @ 4°C	100 ml	14 days
Ammonia	500 ml Plastic	Store @ 4°C, H2SO4	500 ml	28 days
BOD	500 ml Plastic	Store @ 4°C	500 ml	48 hours
Chloride	250 ml Plastic	Store @ 4°C	100 ml	28 days
Chlorine Residual	500 ml Plastic	Store @ 4°C	500 ml	Immed.
COD	250 ml Plastic	Store @ 4°C, H2SO4	250	28 Days
Cyanide	500 ml Plastic	Store @ 4°C NaOH	500	14 Days
Fluoride	250 ml Plastic	Store @ 4°C	200 ml	28 days
Hardness – Total as CaCO3	250 ml Plastic	Store @ 4°C	250 ml	6 months

EPA Method	Sample Container ¹	Sample Preservation	Preferred Volume	EPA Holding Time ⁴
Nitrate	250 ml Plastic	Store @ 4°C	250 ml	48 hours
Nitrite	250 ml Plastic	Store @ 4°C	250 ml	48 hours
Perchlorate	250 ml Plastic	Store @ 4°C	250 ml	28 days
Divalent Manganese	40 ml VOA	Store @ 4°C	40 ml	14 days
Volatile Fatty Acids	40 ml VOA	Store @ 4°C	40 ml	14 days
Oil & Grease	1 L Glass	Store @ 4°C, H2SO4	1000 ml	28 days
Oxygen Dissolved	1 L Glass Bottle and Top	Store @ 4°C	500 ml	Immed.
Phosphate – Ortho	250 ml Plastic	Store @ 4°C	100 ml	48 hours
Residue, Total (TS)	250 ml Plastic	Store @ 4°C	250 ml	7 days
Residue, Filterable (TDS)	250 ml Plastic	Store @ 4°C	250 ml	7 days
Residue, Nonfilterable (TSS)	250 ml Plastic	Store @ 4°C	250 ml	7 days
Residue, Settleable (SS)	1 L Plastic	Store @ 4°C	1000 ml	48 hours
Specific Conductance	250 ml Plastic	Store @ 4°C	100 ml	28 days
Sulfate	250 ml Plastic	Store @ 4°C	100 ml	28 days
Sulfide	500 ml Plastic	Store @ 4°C, NaOH pH>9, Zn Acetate	500 ml	7 days
Turbidity	250 ml Plastic	Store @ 4°C	100 ml	48 hours

Notes for Table 5:

- 1. VOA (volatile organic analysis) vial 40 ml with Teflon-faced silicone cap liner. Samples are collected with no headspace.
 - 1L Amber, a one liter amber glass bottle with Teflon-lined screw cap. If amber bottles are not available, wrap the bottle in aluminum foil to protect from light.

2. If the sample contains free or combined chlorine, add sodium thiosulfate (10 mg/40 ml) to the VOA vial before collecting the sample. For the 1L amber glass bottle, add 80 mg sodium thiosulfate per liter of sample.

- 3. If the water sample is to be stored for more than 7 days before analysis, add HCl to adjust the pH to <2.
- 4. 14 days means: Analysis must occur within 14 days of sampling. 7/40 means: 7 days for extraction and 40 days for analysis, depending on sample matrix. Waters are 7 days, soils are 14 days.

6.3 Sample Custody

Chain-of-custody procedures have been established to document the identity of a sample and its handling from the time of collection until its ultimate disposal. A chain-of-custody must accompany all samples.

The sampling technician in the field initiates a chain-of-custody record which remains with the sample throughout its handling from the field collection, to delivery to the laboratory, to analysis in the laboratory. A sample chain-of-custody record is shown in Figure 1.

Verification of sample integrity is one of the main responsibilities of the sample control officer. The sample is inspected to ensure that:

- The sample is clearly marked and sampling date is included.
- The sample was collected in an appropriate container for the analysis.
- The sample is properly preserved.
- There is sufficient volume to do all the analyses required.
- Samples should match those in the chain-of-custody

If the above conditions are met, the sample is then assigned a unique log number which, in addition to being attached to the sample container, is entered on the chain-of-custody record, in the sample log book, and into the computerized data handling system (electronic LIMS). Besides the project and log numbers, the computerized record also contains the client name, the sample description, the sample matrix type, the required analytical parameters, and the report due date. All records of received materials are maintained.

If the above conditions are not met, the client who originally brought the sample to the laboratory must be contacted. Option can be given as to what can be done with the samples as is or if new samples are required.

Figure 2. Chain-of-Custody Sample Form

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Contains Date Time Type Contains Number Contains C	Address						Test /					_	Test Requirements
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6.4 Sample Handling

After samples have been logged in, the samples (or subsamples) are refrigerated at 4°C. VOA vials are separated from other samples to prevent vapor-phase cross contamination. If aliquots or subsamples are to be split out of a sample, care is taken to ensure that the subsamples are representative of the original. Blending or grinding may be required.

American Analytics follows the general procedures for sample handling for trace organic analysis as discussed in Chapter 8 of the "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" (EPA, 1979). Specific procedures for sample handling are given in EPA Manual SW-846 or in the Federal Register, and are summarized in Table 4 for the methods performed by American Analytics. Field personnel are informed of the guidelines for sample collection and container labeling. They are also informed of the policy governing acceptance or rejection of samples delivered for analysis. All samples and extracts are tracked from receipt, storage, handling, analysis, reporting, and disposal by their unique American Analytics sample number.

Samples are available to personnel within the laboratory who need access. Refrigerators are centrally located. Sample controllers provide some sample security and only authorized laboratory personnel may store and/or handle samples in the laboratory.

7.0 CALIBRATION PROCEDURES

All reagent chemicals used by American Analytics are of ACS reagent grade or better, purchased from reputable laboratory supply companies. Standards are prepared in the laboratory from high-purity starting materials or purchased as standard concentrates.

Calibration procedures differ by analytical method (refer to Table 6 for specific details.)

7.1 Instrument Calibration

7.1.1 Volatile Organics by GC/MS

The instrument tune is checked with BFB (bromofluorobenzene) every twelve (12) hours of operation. Specific ions resulting from electron impact fragmentation must meet EPA specified ion abundance criteria. The initial calibration of the GC/MS is conducted as necessary, using standards prepared at five different concentrations. Response factors of the System Performance Check Compounds (SPCC's) must exceed 0.300. The percent relative standard deviation of the Cali-

bration Check Compounds (CCC's) must be less than or equal to 30%. Percent relative standard deviation of non-CCC's must be less than or equal to 15%.

A continuing calibration check is analyzed every twelve (12) hours. The SPCC response factors must be greater than 0.300, and the CCC response factors may not deviate more than 20% from the average response factor of the initial calibration. The internal standard calibration method is used to quantitate samples.

7.1.2 Base/Neutral and Acid Extractable Organics by GC/MS

The instrument tune is checked with DFTPP (decafluorotriphenylphosphine) every twelve (12) hours of operation. Specific ions resulting from electron impact fragmentation must meet EPA specified ion abundance criteria. The initial calibration of the GC/MS is conducted as necessary, using standards prepared at five different concentrations. Response factors of the System Performance Check Compounds (SPCC's) must exceed 0.050. The percent relative standard deviation of the Calibration Check Compounds (CCC's) must be less than or equal to 30%. The percent relative standard deviation of non-CCC's must be less than or equal to 15%.

A continuing calibration check is analyzed every twelve (12) hours. The SPCC response factors must be greater than 0.050, and the CCC response factors may not deviate more than 20% from the average response factor of the initial calibration. The internal standard calibration method is used to quantitate samples.

7.1.3 Volatile Organics by Gas Chromatography

Initial calibration is performed with a minimum five (5) standards prepared at five different concentrations. The percent relative standard deviation of the response factors for each analyte calculated at each of the five concentration levels must not exceed 20%. Prior to sample analysis a calibration verification standard is run to verify the validity of the calibration. The percent difference between the average response factor of the initial standard curve for a specific parameter and the response factor of the continuing calibration for the same parameter must be within +/- 15%. If this criterion is not met for all parameters then the average of the responses for all parameters must be within +/- 15%.

7.1.4 Semi-Volatile Organics by Gas Chromatography

Initial calibration is performed with a minimum five (5) standards prepared at five different concentrations. The percent relative standard deviation of the response factors for each analyte calculated at each of the five concentration levels must not exceed 20%. Prior to sample analysis a calibration verification standard is run to verify the validity of the calibration. The percent difference between the average

response factor of the initial standard curve for a specific parameter and the response factor of the continuing calibration for the same parameter must be within +/- 15%. If this criterion is not met for all parameters then the average of the responses for all parameters must be within +/- 15%.

7.1.5 Metals by ICP

Each day prior to sample analysis, an instrument calibration is performed. The calibration is then verified by analyzing the Initial Calibration Verification (ICV) standard which must lie within 10% of the true concentration. Following the ICV, a Continuing Calibration Verification (CCV) standard is analyzed every ten (10) samples which must also lie within 10% of the true concentration. Standards are prepared by diluting mixed-element concentrates, which are themselves prepared from commercially available solutions. Comparability studies are carried out frequently to validate the concentrations of the commercial standards.

7.1.6 Metals by Graphite Furnace and Flame

Each day prior to sample analysis, an instrument calibration is performed using a minimum of three (3) standards. The calibration curve must have a correlation coefficient of 0.995 or greater. Following the initial calibration, a Continuing Calibration Verification (CCV) is analyzed every ten (10) samples. The apparent concentration of the CCV must lie within 10% of the true concentration. Standards are prepared by diluting mixed-element concentrates, which are themselves prepared from commercially available solutions. Comparability studies are carried out frequently to validate the concentrations of the commercial standards.

7.1.7 Colorimetric Analyses

Nitrate, nitrite, ferrous iron, COD, cyanide and phosphates fall into this category. A calibration curve of at least three standards is prepared daily. The correlation coefficient of the curve must be 0.995 or greater.

7.1.8 Titrimetric Analyses

Hardness, alkalinity, chloride, and calcium fall into this category. Titrants are standardized every three months with primary standards.

7.1.9 Gravimetric Analyses

Oil and grease, dissolved solids, and suspended solids fall into this category. Each analysis depends heavily on the accuracy of the balance used. For this

reason, balances are calibrated annually and checked on a weekly basis with class "S" weights. The recorded weight must agree within 0.1% of the expected weight.

7.1.10 pH

The pH meter is calibrated with two buffers separated by three pH units prior to analysis each day. The reading must be within 0.1 unit of the true value.

7.1.11 <u>BOD</u>

An ambient air calibration of the DO meter is performed daily prior to sample analysis. The calibration is also verified by taking a DO measurement of laboratory reagent water saturated with DO. The DO concentration must fall within +/- 15% of the literature value of water saturated with DO at the temperature at which the measurement is taken.

Table 6
Calibration Procedures

Reference	Analysis	Calibration	Frequency	Acceptance
AA SOP#		Methods		Criteria
		General Che	mistry	
IC01	Anions by IC Nitrate Nitrite Sulfate Phosphate Chloride	Calibration Curve Continuing Calibration	As needed base on CCV Every 10 samples	>= 0.995 correlation +/- 10% of theoretical value
	Bromide			
IC02	Perchlorate by IC	Calibration Curve	As needed base on CCV	>= 0.995 correlation
		Continuing Calibration	Every 10 samples	+/- 10% of theoretical value
IC05	Divalent Manganese by IC	Calibration Curve	As needed base on CCV	>= 0.995 correlation

Reference	Analysis	Calibration	Frequency	Acceptance
AA SOP#		Methods		Criteria
		Continuing Calibration	Every 10 samples	+/- 10% of theoretical value
IC06	Volatile Fatty Acids by IC	Calibration Curve	As needed base on CCV	>= 0.995 correlation
		Continuing Calibration	Every 10 samples	+/- 10% of theoretical value
IC03	Hexavalent Chromium By IC	Calibration Curve	As needed based on CCV	>= 0.999 correlation
		Continuing Calibration	Every 10 samples	+/- 10% of theoretical value
GEN22	Oil and Grease by IR	Calibration Curve	As needed based on CCV	>= 0.995 correlation
GEN22	Total Recoverable Petroleum Hydrocarbons	Continuing Calibration	Every 20 samples	+/- 20% of theoretical value
GEN01	pH	Two buffers	Daily	Within 0.1 unit of true value
GEN09	Nitrite	Calibration Curve	As needed base on CCV	>= 0.995 correlation
GEN10	Nitrate			
GEN13	COD	Continuing Calibration	Every 20 samples	+/- 10% of theoretical value
GEN21	Cyanide			

Reference	Analysis	Calibration	Frequency	Acceptance
AA SOP#	·	Methods		Criteria
GEN03	Total Dissolved Solids	Balance Check	Weekly	Within 0.1% of expected value
GEN04	Total Suspended Solids Oil and	Balance Service and calibration	Annually	
GEN16	Grease, Gravimetric	and campration		
		Elemental Ar	nalysis	
MET01	ICP Metals (Al, Ba, Ca, Cr, Cu, Pb, Mn, Ni,	Calibration curve	Each batch	>= 0.995 correlation
	TI, Sb, Be, Cd, Co, Fe, Mg, Mo, K, Na, V,	Calibration blank	Every 10 samples	within 3 std dev. of the mean
	Zn)	Continuing calibration	Every 10 samples	+/- 10% of the theoretical value
MET02	Flame Metals	Calibration curve	Each batch	>= 0.995 correlation
	(Al, Be, Cr, Fe, Mn, K, V, Sb, Ca, Co, Pb, Mo, Na, Zn, Ba, Cd, Cu, Mg, Ni, Tl, Ag)	Continuing calibration	Every 10 samples	+/- 10% of the theoretical value
MET03	Graphite Furnace Metals	Calibration curve	Each batch	>= 0.995 correlation
	(As, Be, Cr, Cu, Se, Tl, Sb, Cd, Co, Pb, Ag)	Continuing calibration	Every 10 samples	+/- 10% of the theoretical value
MET07	Cold Vapor Metals (Hg)	Calibration curve	Daily	>= 0.995 correlation
		Organic Ana	llyses	
GC01	Volatile Organics by GC	Calibration curve (5 pt minimum)	As needed based on CCV results	Percent RSD <=20

Reference	Analysis	Calibration	Frequency	Acceptance
AA SOP#		Methods		Criteria
		Continuing calibration standard	Each batch	Specific to each method (refer to SOP)
GC05 GC06	Semi-Volatile Organics (Organochlori ne Pesticides and PCB's) by GC	Calibration curve (5 pt minimum) Continuing	As needed based on CCV results Each batch	Percent RSD <=20
		calibration standard	Lacification	Specific to each method (refer to SOP)
HP01 HP02	Polynuclear Aromatic Hydrocarbons (PAHs) by HPLC	Calibration curve (5 pt minimum)	As needed based on CCV results	Percent RSD <=20
	HFLG	Continuing calibration standard	Each batch	Specific to each method (refer to SOP)
MS01 MS02	Volatile Organics by GC/MS	Tune check w/BFB	Every 12 hours of operation	Ion abundance criteria(see SOP)
		Initial calibration	As needed based on CCV results(CCC's and SPCC's)	SPCC's with RF>= 0.300; CCC's %RSD<= 30; Non CCC's %RSD <=15
		Continuing calibration	Every 12 hours of operation	SPCC's with RF>= 0.300; CCC's RF deviates < =20% from average of initial calibration
MS09	Volatile Organics in Drinking Water	Tune check w/BFB	Every 12 hours of operation	Ion abundance criteria (see SOP)
		Initial calibration	As needed based on CCV	%RSD≤20 for all analytes

Reference	Analysis	Calibration	Frequency	Acceptance
AA SOP#		Methods		Criteria
		Continuing calibration	Every 12 hours of operation	%Diff≤30 for all analytes
MS03 MS05	Semi-Volatile Organics by GC/MS	Tune check w/BFB	Every 12 hours of operation	Ion abundance criteria(see SOP)
		Initial calibration	As needed based on CCV results(CCC's and SPCC's)	SPCC's with RF> 0.050; CCC's %RSD<= 30; Non CCC's %RSD <=15
		Continuing calibration		
			Every 12 hours of operation	SPCC's with RF>= 0.050; CCC's RF deviates <= 20% from average of initial calibration

7.2 Standards Preparation and Calibration Procedures

7.2.1 Standard Preparation

Commercially prepared and certified stock standard solutions are used, if they are available, from suppliers such as Supelco, Accustandard or Ultra Scientific.

Otherwise, the standards are prepared in the laboratory, using reagent grade chemicals. Working solutions are prepared by diluting the stock solutions accordingly.

The standard preparation procedures are documented in detail in the method specific laboratory Standard Operating Procedures (SOP's). Information regarding the standard preparation date, lab identification code, concentration, vendor, lot numbers, expiration date and other details are documented in the appropriate standard preparation log book. All neat organic compounds are entered into an inventory system which includes recording the following: purity, date received, supplier, supplier stock number, lot number and storage location. Stock and working solutions are traceable to the parent compound. When a new standard is

prepared, its response is compared with that of the replaced standard to check for gross errors. Standards from commercial sources are replaced no later than the expiration date supplied by the manufacturer or one year, whichever is sooner. When determining a standard replacement schedule, consideration must be made for stability and volatility of the solvent as well as the analyte.

7.2.2 External Standard Calibration Procedure

For each analyte of interest, prepare calibration standards at three to five concentration levels as specified in the SOP by adding volumes of one or more stock standards to a volumetric flask, and diluting to volume with an appropriate solvent. One of the external standards should be at a concentration near, but above, the method detection limit. The other concentrations should correspond to the expected range of concentrations found in real samples, or should define the working range of the detector.

Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (e.g., 1- to 5-µL injections, purge-and-trap, etc.) Tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each analyte. Alternatively, for samples that are introduced into the gas chromatograph using a syringe, the ratio of the response to the amount injected, defined as the calibration factor (CF), can be calculated for each analyte at each standard concentration. For analytical methods using the external standard calibration procedure if the percent relative standard deviation (%RSD) of the calibration factor is less than 20% over the working range, linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve.

Calibration Factor =
$$\frac{Area\ of\ Peak}{Mass\ injected\ (in\ nanograms)}$$

Percent Difference =
$$\frac{R_1 - R_2}{R_1} \times 100$$

Where:

 R_1 = Calibration factor from first analysis.

 R_2 = Calibration factor from succeeding analyses.

$$\%RSD = \frac{S}{\overline{X}} \times 100$$

Where:

S = Standard deviation of calculated calibration factors.

X = Arithmetic mean of calculated calibration factors.

7.2.3 Internal Standard Calibration Procedure

To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Due to these limitations, no internal standard applicable to all samples can be suggested.

Tabulate the peak height or area responses against the concentration of each compound and internal standard. Calculate response factors (RF) for each compound as follows:

$$RF = (A_sC_{is})/(A_{is}C_s)$$

Where:

 A_s = Response for the analyte being measured.

 A_{is} = Response for the internal standard.

 C_{is} = Concentration of the internal standard.

 C_s = Concentration of the analyte being measured.

The RF must meet method specific criteria for each parameter of interest as stated in the Standard Operating Procedure (SOP).

7.2.4 Method of Standard Additions

Equal portions of sample are added to a water blank and standard. For more accuracy multiple additions of sample are made. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated back to zero absorbance, the point of interception of the abscissa is the concentration of the unknown.

8.0 STANDARD OPERATING PROCEDURES

Methods specified in the Federal Register, October 26, 1984 and in EPA Manual SW-846 are the basis for Standard Operating Procedures (SOP) used by American Analytics. An SOP is present for each analysis performed by American Analytics. These SOPs are kept in the laboratory for use by the analysts.

Standard Operating Procedures may only be changed by the QA/QC officer following final approval by the Laboratory Director.

9.0 DATA RECORDING, REDUCTION, VALIDATION, AND REPORTING

9.1 Data Recording

All analytical procedures are recorded in laboratory notebooks. This includes the method used, the sample identity, any variations from the standard procedure, dilution of the sample, volumes used, etc. The originals of all records and notebooks are kept on file in the laboratory. Clients may receive copies of the records, along with the official report that is submitted. Other records are kept separately from the laboratory in the administrative files.

9.2 Data Reduction

Most data produced in the laboratory are generated through the use of dedicated instrumentation with microcomputer interfaces. These PC-based systems receive the original signal from the instrument to which the sample or extract was submitted. The PC--or, for some larger instruments, a dedicated minicomputer-transforms the raw signal into a quantitative value.

An experienced analyst reviews this "candidate" result either on screen or on a printout, verifying identifications, double-checking quantitative formulas, and acquiring final numerical values. The analyst writes out calculated results or checks off computer-produced results directly on the computer printout. The printout is cross-referenced to a file number in a bound run log.

Some instruments are configured to operate without computers. For these, the signal is recorded as a strip-chart trace, as numerical output on a printer strip, or as a direct reading from a digital or analog dial. In such cases, the analyst must then reduce the data to a reportable format, multiplying the original signal by a calibration factor or comparing it with a standard curve. Blank correction may be required. The aliquot result must be divided by the mass or volume of the sample to produce a concentration-based final result. Simple programs are used for some calculations; most are carried out on handheld scientific calculators. All these data are recorded in a bench book for the particular determination in question. The analyst enters results for single or multiple component tests in the assigned book by hand.

Some lab tests, such as titrations or sensory evaluations, do not use instruments. For these, the analyst records the quantitative result or observation directly in a bound book. The same calculations as those described in the last paragraph may be needed; if so, they are recorded in the book.

Raw data with accompanying calculations are maintained in individual project files for future reference.

9.3 Data Validation

After data reduction has occurred, draft reports of the analytical results are submitted to the operations manager for review. The raw data and calculations are reviewed to ensure that the chemists correctly interpreted the data and did not make errors in the calculation of the reported analytical results. The operations manager also reviews the data to ensure the all QA/QC requirements are met. For each analytical method the analytical batch is checked to ensure that all QA/QC elements were performed. The QA/QC results are checked to identify potential deviations from the method QA/QC acceptance criteria. If deviations are found, the appropriate corrective action (see section 13.0) must be taken before the results can be released.

9.4 Reporting

After the data is reviewed, the final report is printed automatically by use of the Laboratory Information Management System (LIMS). An example page of the analytical report can be seen in Figure 2. If there is a client inquiry prior to completion of all analytical work on a specific project, a partial report of analytical results can be printed by the LIMS. Supplemental items can also be generated by the LIMS and submitted to the client such as:

- QA/QC reports in various formats
- Tabulated chronological trending of analytical results
- Results in electronic format (Excel, DBF, ASCII comma and quote delimited and various other formats.

When the final report is printed, it is reviewed and signed by the project manager.

Final reports are stored in their respective project files within the laboratory.

Figure 3. Example of Analytical Report Format



LABORATORY ANALYSIS RESULTS

Page 1

Client: Project No.: N/A Project Name: Sample Matrix: Water Method: EPA 8020 (BTEX) AA Project No.: A135218-17 Date Received: 02/11/00 Date Reported: 02/23/00

Units: ug/L

Date Sampled:	02/10/00	02/10/00	02/10/00	02/10/00	
Date Analyzed: AA ID No.: Client ID No.:	02/16/00 102247 MW-6	02/16/00 102248 MW-4	02/16/00 102249 MW-7	02/16/00 102250 MW-5	MRL
Compounds:					
Benzene	< 0.3	<0.3	0.50	0.40	0.3
Ethylbenzene	< 0.3	< 0.3	< 0.3	< 0.3	0.3
Toluene	< 0.3	< 0.3	< 0.3	< 0.3	0.3
Xylenes	< 0.5	< 0.5	< 0.5	0.50	0.5

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10.0 INTERNAL QUALITY CONTROL CHECKS

10.1 Continuing Calibration Standards

Continuing calibration standards are used to verify the validity of an instrument or method calibration and dictate their ability to accurately quantify the concentration of a target parameter in an unknown sample. The chemical compounds that must be used in the calibration of each method are listed in the appropriate SOP. The results generated from the analysis of a continuing calibration standard must lie within specific acceptance criteria otherwise corrective action must be taken which may require instrument or method re-calibration.

10.2 Method Blanks

Method blanks for aqueous samples consist of organic-free or deionized water carried through the analytical scheme like a sample. For solid matrices method blanks are prepared using Ottawa sand to simulate solid matrix effects. Method blanks serve to measure contamination associated with laboratory storage, preparation, or instrumentation. For most tests, one method blank is analyzed in every analytical batch of samples.

10.3 Travel Blanks and Field Blanks

Travel and field blanks identify contamination that occurs during sample transportation or collection. Travel blanks originate in the laboratory, where a sample vial is filled with organic-free reagent water and carried with other sample containers out to the field and then back to the lab. Field blanks originate as empty sample vials, which are carried to the sampling site and filled with organic-free water at the location.

10.4 Sample Blanks

Sample blanks are used when characteristics such as color or turbidity interfere with a determination. In a spectrophotometric method, for example, the natural absorbency of the sample is measured and subtracted from the absorbency of the developed sample. Sample blanks are run only as necessary.

10.5 Calibration Blanks

Calibration blanks are prepared with standards to create a calibration curve. They differ from other standards only by the absence of an analyte, and provide the "zero-point" for the curve.

10.6 Internal Standards

Internal standards are measured amounts of certain compounds added after sample preparation or extraction. They are used in an internal standard calibration method to correct sample results suffering from capillary column injection losses, purging losses, or the effects of viscosity. internal standard calibration is currently used for volatile organics, semi-volatile organics, GC/MS extractables, and metals by ICP.

10.7 Surrogates

Surrogates are measured amounts of certain compounds added before sample preparation or extraction. Analysts measure the recovery of the surrogate to determine systematic extraction problems. Surrogates are added to all samples analyzed for chlorinated pesticides, GC/MS extractables, volatiles, and GC volatiles.

10.8 Spikes

Spikes are aliquots of samples to which known amounts of an analyte have been added. Stock solutions used for spiking are purchased or prepared independently of calibration standards. Prepared and analyzed in each batch of samples, spikes are subjected to the same preparation/extraction procedure and analysis as the samples in question. Spike recovery measures the effects of interferences in the sample matrix and reflects the accuracy of the determination. Spike recoveries are calculated as follows:

P = 100 (A - B)/T

Where:

P = percent spike recovery

A = concentration determined in spiked sample

B =concentration determined in original unspiked sample

T = true value of spike added.

The accuracy of each method is assessed by maintenance of records on matrix spike analysis. After the first five spike samples have been analyzed, the average P_a and standard deviation S_p of the percent recovery for each spike compound are calculated. The accuracy assessment is expressed as a percent recovery interval from P_a - $2S_p$ to P_a + $2S_p$. The values of P_a and S_p are updated after each five to ten measurements. In addition, all spike recovery measurements after the first five are plotted on control charts, as percent recovery vs. analysis number. (Control charts are described in detail in Section

12.3 of this manual.) Any measurement higher than P_a+2S_p or lower than P_a-2S_p serves as a warning that the analytical system may be out-of-control.

10.9 Duplicates and Duplicate Spikes

Duplicates are additional aliquots of a sample that are subjected to the same preparation-and analytical scheme as the sample. When the analyte concentration is consistently below the detection limit, duplicate spikes are substituted for duplicates. Duplicates and duplicate spikes are prepared and analyzed in every batch basis of samples. The relative percent difference (RPD) between duplicates or duplicate spikes measures the precision of a given analysis and is calculated as follows:

$$RPD = [(R1 - R2)/Rav]X100$$
 (or)= $[(S1 - S2)/Sav]X100$

where R1 and R2 = duplicate determinations of the analyte in the sample S1 and S2 = observed concentrations of analyte in the spike and its duplicate Rav = average determination of the analyte concentration in the original sample Sav = average of observed analyte concentrations in spike and its duplicate

10.10 Laboratory Control Standards

Laboratory control standards (LCS's)--or quality control check standards (QCCS's)—for aqueous samples are aliquots of organic-free or deionized water to which known amounts of an analyte have been added. For solid matrices, LCSs are prepared using Ottawa sand to which known amounts of an analyte have been added. Sand is used to simulate solid matrix effects. The LCSs are subjected to the same preparation/ extraction procedure and analysis as samples. Stock solutions used for LCS's are purchased or prepared independently of calibration standards. LCS recovery tests the functioning of analytical methods and equipment.

LCS's are prepared and analyzed with every batch of samples. The true value and recovered concentration are archived and retrievable for statistical analysis. Laboratory control limits are calculated when 30 data points become available. Control charts for LCS's can be generated on demand.

11.0 PERFORMANCE AND SYSTEM AUDITS

11.1 Performance Audits

Performance audits are carried out to quantitatively evaluate the measurements made by the laboratory on a regular basis.

The audits are carried out under the supervision of the QA/QC officer. The QA/QC Officer is trained in audit performance. Training is performed by other senior laboratory personnel with experience in auditing. External reference samples are analyzed bi-annually to audit the performance of the analytical procedures, particularly to assess the accuracy of the measurements. These audits are performed for each method carried out by the laboratory. The audit samples are not identified, and are carried through the analytical procedure exactly the same as normal samples. The reference samples are obtained from commercial suppliers.

11.2 System Audits

A system audit is a qualitative evaluation of all components of the laboratory quality control measurement system. The laboratory is audited quarterly by the laboratory QA/QC officer. This systems audit includes evaluation of the analytical instruments, personnel, facilities, adherence to the method procedures, and quality control. Examples of questions addressed by the QA/QC officer during a system audit are shown in Appendix B.

Clients are provided access, by request, to our laboratory to perform their own audits as well.

11.3 Review of Analytical and Quality Control Results

The project manager reviews both the sample data and the quality control data. This review covers 100% of the analyses which are performed. Quality control is monitored through the use of control charts, as described in Section 12.3.

Any incorrect or out-of-control situations which are detected by the supervisor are corrected before analysis is allowed to continue. Corrective actions are described in Section 13.0.

All analytical and quality control results and corrective action procedures must be reviewed and approved by a supervisor and the QA officer. Approval must be indicated by their signature.

12.0 ROUTINE PROCEDURES TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS

12.1 Precision

Precision is determined by duplicate analyses, as previously described in Section 5.0. Precision is calculated as the relative percent difference (RPD) between duplicate samples:

$$RPD = \frac{-X_A - X_B}{X_m} \times 100$$

Where X_A and X_B are values from duplicate analyses and X_m is the mean value of X_A and X_B . Values of RPD are calculated, and plotted on a control chart, as described in Section 12.3.

If the control limits for precision are exceeded, then corrective action must be taken before the analysis is completed. The upper and lower control limits and warning limits for precision are defined as follows:

Upper Control Limit, UCL = RPD + 3s Lower Control Limit, LCL = RPD - 3s Upper Warning Limit, UWL = RPD + 2s Lower Warning Limit, LWL = RPD - 2s

Where the standard deviation s of the RPD is:

$$s = \sqrt{\frac{\sum (RPD)^2 - (\sum RPD)^2 / n}{n - 1}}$$

Where n is the number of duplicate pairs evaluated for the parameter in question.

12.2 Accuracy

Accuracy is defined for spiked samples as the percent recovery P:

$$P = 100 \; \frac{A - B}{T}$$

Where A is the measured spike sample value, B is the sample value before spiking (background), and T is the value of the spike which was added to the sample.

The average recovery P_a is calculated as:

$$P_a = \sum P_i / n$$

Where i ranges from 1 to n, n is the number of spike samples and P_i is the recovery of the spiked analyte in each sample.

The upper and lower control and warning limits are:

$$\begin{array}{ll} UCL = & P_a + 3S_p \\ LCL = & P_a - 3S_p \\ UWL = & P_a + 2S_p \\ LWL = & P_a - 2S_p \end{array}$$

Where the estimated standard deviation of the recovery S_p is calculated as:

$$S_p=[\sum (P_i-P_a)^2]^{1/2}$$
 Where: i ranges from 1 to n

Values of percent recovery P are plotted on control charts, as described in Section 12.3.

12.3 Control Charts

Control charts are used to monitor the precision and accuracy of the analytical methods, and to determine whether the QC data are within control limits. The construction and use of control charts are described in Reference 2, and in Chapter 7 of Reference 3.

A control chart shows how a measured quantity compares with previous measurements of that quantity. Control and warning limits are calculated as described in Section 10.3. Information is compiled and incorporated into the control charts by the QA/QC Officer on a monthly basis.

If the data exceeds the control limits, (i.e., if the measured value is more than three standard deviations away from the previously established average), then corrective action must be taken before the results are reported. If the data exceeds the warning limits, (i.e., if the measured value is more than two but less than three standard deviations away from the previously established average), then the system should be monitored for possible corrective action. The control and warning limits are recalculated periodically, after every 20 measurements or once a year, whichever is more frequent.

13.0 CORRECTIVE ACTION

Quality control failures logically fall into two categories:

- Single QC outliers
- Systematic failure

QC outliers are identified by comparing the results from the analysis of the QA/QC samples to the control limits established for each method. Analytical control limits are maintained for laboratory control standards (LCS's), method blanks, spike recoveries, duplicates, and surrogate recoveries. In addition, many analytical methods have quality control criteria for calibrations, sensitivity checks, and other method-specific quality checks that are performed routinely. The acceptance limits for most QA/QC criteria are based on historical data collected in the laboratory and are revised periodically.

If one of the above checks does not meet the acceptance criteria, the analyst at the bench, and sometimes the section supervisor, initiates corrective action. Such action is initiated by documenting the failure, identifying the source of the problem and deciding on a course of action to correct the problem. Once the source of the problem is identified, implementation of the corrective action is usually quick with little interruption in analysis. The nature of the problem, corrective action, and the result are documented in the laboratory corrective action form.

Systematic failures of a method, issues of method compliance, consistent contamination that the analyst cannot resolve, QC issues raised in audit reports, or QC issues that impact data already reported, are examples of more serious problems that are dealt with directly by the laboratory QA/QC officer who with the assistance of management and other technical staff in the laboratory identify the appropriate corrective action that will solve the problem. All details of this process are formally documented for future reference.

Table 7
Summary of Corrective Action Procedures

Reference	Analysis	Control	Acceptance	Corrective		
AA SOP#		Item	Criteria	Action		
	General Chemistry					
IC01	Anions by IC Nitrate Nitrite	Calibration Curve	>= 0.995 correlation	Rerun calibration standards		
	Sulfate Phosphate	Continuing Calibration	+/- 10% of theoretical value	Recalibrate, rerun last 20 samples		
	Chloride Bromide	Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch		
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze		
		Duplicate	Above 10 X RDL; %RPD must be within current control limits	Reprep to confirm matrix interference		
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze		
IC03	Hexavalent Chromium By IC	Calibration Curve	>= 0.999 correlation	Rerun calibration standards		
		Continuing Calibration	+/- 10% of theoretical value	Recalibrate, rerun last 10 samples		
		Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch		
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze		

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
		Duplicate	Above 10 X RDL; %RPD must be ≤20%.	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
GEN22	Oil and Grease by IR	Calibration Curve	>= 0.995 correlation	Rerun calibration standards
	Total Recoverable Petroleum	Continuing Calibration	+/- 20% of theoretical value	Recalibrate, rerun last 20 samples
	Hydrocarbons	Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate, Duplicate spike	Above 10 X RDL; %RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
GEN01	рН	Two buffers	Within 0.1 unit of true value	Recalibrate instrument Rerun to confirm
		Duplicate	% RPD must be within current control limits	matrix interference
		Laboratory control		Solve problem and

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
		standard	Within current control limits	reanalyze batch
GEN09	Nitrite	Calibration Curve	>= 0.995 correlation	Rerun calibration standards
GEN10	Nitrate			
GEN13	COD	Continuing Calibration	+/- 10% of theoretical value	Recalibrate, rerun last 20 samples
GEN21	Cyanide	Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate	Above 10 X RDL; %RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
GEN03	Total Dissolved Solids	Balance Check	Within 0.1% of expected value	Recalibrate balance
GEN04	Total Suspended Solids	Method Blank	Less than 5X reporting detection	Solve Problem; Reanalyze batch
GEN16	Oil and Grease, Gravimetric	Spiked sample	limit Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate, Duplicate spike	Above 10 X RDL; %RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control	Within current	Examine spike; if

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
		standard	control limits	spike also out, reprep batch and reanalyze
	Elemental A			
MET01	ICP Metals (Al, Ba, Ca, Cr,	Calibration Curve	>= 0.995 correlation	Rerun calibration standards
	Cu, Pb, Mn, Ni, Tl, Sb, Be, Cd, Co, Fe, Mg, Mo, K, Na, V, Zn)	Calibration blank	Within 3 standard deviation of mean	Rerun blank
		Continuing Calibration	+/- 10% of theoretical value	Recalibrate, rerun last 20 samples
		Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate, Duplicate spike	Above 10 X RDL; %RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
MET02	Flame Metals (Al, Be, Cr, Fe,	Calibration Curve	>= 0.995 correlation	Rerun calibration standards
	Mn, K, V, Sb, Ca, Co, Pb, Mo, Na, Zn, Ba, Cd, Cu, Mg, Ni, Tl, Ag)	Calibration blank	Within 3 standard deviation of mean	Rerun blank
		Continuing Calibration	+/- 10% of theoretical value	Recalibrate, rerun last 20 samples

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
		Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate, Duplicate spike	Above 10 X RDL; %RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
MET03	Graphite Furnace Metals	Calibration Curve	>= 0.995 correlation	Rerun calibration standards
	(As, Be, Cr, Cu, Se, Tl, Sb, Cd, Co, Pb,	Continuing Calibration	+/- 10% of theoretical value	Recalibrate, rerun last 20 samples
	Ag)	Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
MET07	Cold Vapor Metals (Hg)	Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate, Duplicate spike	Above 10 X RDL; %RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
		Organic Ana	llyses	
GC001	Volatile Organics by GC	Calibration Curve	%RSD <= 20	Rerun calibration standards
		Continuing Calibration	Within limits dictated in method specific SOP	Rerun cont. calibration; recalibrate if still out
		Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate spike	%RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
		Surrogate recovery	Within current control limits	Reprep and reanalyze sample
			0/DCD 00	Damus as Physics
GC05 GC06	Semi-Volatile Organics (Organochlori	Calibration Curve	%RSD <= 20	Rerun calibration standards
	ne Pesticides and PCB's) by GC	Continuing Calibration	Within limits dictated in method specific SOP	Rerun cont. calibration; recalibrate if still out

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
		Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate spike	%RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
		Surrogate recovery	Within current control limits	Reprep and reanalyze sample
HP01	Polynuclear	Calibration Curve	%RSD <= 20	Rerun calibration
HP02	Aromatic Hydrocarbons (PAHs) by HPLC	Continuing Calibration	Within limits dictated in method specific SOP	standards Rerun cont. calibration; recalibrate if still out
		Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate spike	%RPD must be within current control limits	Reprep to confirm matrix interference
			Within current control limits	Examine spike; if spike also out, reprep

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
		Laboratory control standard	Within current control limits	batch and reanalyze Reprep and reanalyze sample
		Surrogate recovery		
MS01 MS02	Volatile Organics by GC/MS	Tune Check with BFB	Ion abundance criteria (See SOP)	Tune instrument; repeat
		Calibration Curve	SPCC's with RF> 0.300; CCC's %RSD< 30; Non CCC's %RSD <15	Recalibrate instrument
		Continuing Calibration	SPCC's with RF> 0.300; CCC's RF deviates < 20% from average of initial calibration	Rerun cont. calibration; recalibrate if still out
		Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate spike	%RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
		Surrogate recovery	Within current control limits	Reprep and reanalyze sample

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
MS09	Volatile Organics in Drinking Water by	Tune Check with BFB	Ion abundance criteria (See SOP)	Tune instrument; repeat
	GC/MS	Calibration Curve	%RSD≤20 for all analytes	Recalibrate instrument
		Continuing Calibration	%Diff≤30 for all analytes	Rerun cont. calibration; recalibrate if still out
		Method Blank	Less than 5X reporting detection limit	Solve Problem; Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate spike	%RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze
		Surrogate recovery	Within current control limits	Reprep and reanalyze sample
MS03 MS05	Semi-Volatile Organics by GC/MS	Tune Check with DFTPP	Ion abundance criteria (See SOP)	Tune instrument; repeat
		Calibration Curve	SPCC's with RF> 0.050; CCC's	Recalibrate instrument

Reference	Analysis	Control	Acceptance	Corrective
AA SOP#		Item	Criteria	Action
			%RSD< 30; Non CCC's %RSD <15	
		Continuing Calibration	SPCC's with RF> 0.050; CCC's RF deviates < 20% from average of initial calibration	Rerun cont. calibration; recalibrate if still out Solve Problem;
		Method Blank	Less than 5X reporting detection limit	Reanalyze batch
		Spiked sample	Within current control limits	Examine LCS; if LCS also out, reprep batch and reanalyze
		Duplicate spike	%RPD must be within current control limits	Reprep to confirm matrix interference
		Laboratory control standard Surrogate	Within current control limits	Examine spike; if spike also out, reprep batch and reanalyze Reprep and reanalyze
		recovery	Within current control limits	sample

14.0 EQUIPMENT

The American Analytics laboratory is equipped with state of the art instrumentation and other laboratory equipment that provide the ability to perform a large variety of environmental and other analytical testing. The equipment is continually upgraded in order to extend to our clients the benefits of newly emerging technology.

Following is a list of the major equipment currently in use at American Analytics.

As new methods are developed and we add new equipment, the above lists and tables are appropriately updated.

Table 8 Laboratory Equipment List

AMERICAN ANALYTICS Laboratory Equipment List Stationary Laboratory

Stationary Laboratory			
Quantity	Description	Model	
	Organics Department		
11	Hewlett Packard Gas Chromatographs	HP 5890	
2	Screening Gas Chromatographs	SRI	
1	High Performance Liquid Chromatograph (HPLC)	HP 1050	
2	Hewlett Packard Mass Spectrometer Detectors	HP 5970	
3	Hewlett Packard Mass Spectrometer Detectors	HP 5971	
1	Varian Saturn 4D GC/MS/MS	Saturn 4D	
4	OI PID/FID Tandem Detectors	OI 4430	
1	OI ELCD Detector	OI 4420	
1	Hewlett Packard FID Detector	N/A	
2	Hewlett Packard ECD Detector	N/A	
1	UV Absorbance Variable Wavelength Detector (VWD)	HP 1050	
1	Programmable Fluorescence Detector (FLD)	HP 1046A	
4	Tekmar Purge and Trap Unit	LSC2000	
4	Tekmar 16 Position Autosampler	ALS2016	
1	Tekmar Purge and Trap Unit	LSC-2	
1	Tekmar 10 Position Autosampler	ALS-10	
1	OI Purge and Trap Unit	OI 4560	
1	OI Purge and Trap Unit	OI 4460	
2	OI 16 Position Autosamplers	OI MPM-16	
3	Hewlett Packard Autoinjectors	HP 7673A	
2	Foxboro Miran Infrared Spectrometers	1A	
1	Tekmar Pulse Sonication Disruptor	TSD-375	
	Inorganics / Metals		
1	Varian Flame AA Spectrophotometer	Spectr AA-20	
1	Varian Graphite Furnace AA Spectrophotometer	Spectr AA-400	
1	ICP Atomic Emission Spectrophotometer	TJA Atomscan 25	
1	ICP Atomic Emission Spectrophotometer	TJA trace	
1	Dionex Ion Chromatograph	DX-500	
1	Dionex Ion Chromatograph	DX-100	
1	Dionex Ion Chromatograph	DX-300	
1	Mulit-Parameter Instrument	WTW Multilab P4	
1	HF Instruments Turbidimeter	DRT100B	
2	pH Meters	N/A	
	Data/Information Management		
5	Hewlett Packard GC/MS Data System	Enviroquant	
1	Gas Chromatography Data Acquisition System	Turbochrom III	
1	Laboratory Information Management System	AA Micro LIMS	
18	IBM Compatible Computer Systems	Gateway / Pentium	
3	Hewlett Packard Laser Jet Printers	4 Plus	

15.0 INSTRUMENT MAINTENANCE AND REPAIR

Instruments are maintained according to the manufacturer's specifications. Major maintenance/repair is performed by or under the direction of the manufacturer's service personnel. Records of instrument checks and maintenance are kept in logbooks. The maintenance log contains the date, analyst, instrument fault (if any), and corrective or preventive maintenance performed.

16.0 REFERENCES

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- 2. Ouchi, Glenn I., <u>Control Charting with Lotus 1-2-3</u>, American Laboratory, <u>19</u>, 82-95, February 1987.
- 3. U.S. Environmental Protection Agency, <u>Handbook for Analytical Quality Control in Water and Wastewater Laboratories</u>, Office of Research and Development, USEPA, Cincinnati, Ohio, 1979.
- 4. U.S. Environmental Protection Agency, <u>Test Methods for Evaluating Solid Waste</u>, Volume 1B: Laboratory Manual, Physical/Chemical Methods, U.S. Government Printing Office, Washington DC, November 1986.
- 5. U.S. Environmental Protection Agency, <u>Test Methods for Evaluating Solid Waste</u>, Final Update III: Laboratory Manual, Physical/Chemical Methods, U.S. Government Printing Office, Washington DC, December 1996.
- 6. U.S. Environmental Protection Agency, <u>Methods for Chemical Analysis of Water and Wastes</u>, EPA-600/4-79-020 (revised March 1983).
- 7. U.S. Environmental Protection Agency, <u>Methods for the Determination of Metals in Environmental Samples</u>, EPA 600-91-010, USEPA Office of Research and Development, Washington DC, 20460, June 1991.
 - 8. U.S. Environmental Protection Agency, <u>Methods for the Determination of Metals in Environmental Samples</u>, <u>Supplement I</u>, EPA-600/R-95/111, Environmental Monitoring Systems Laboratory Office of Research and Development USEPA, Cincinnati, Ohio, 45268, May 1994.

APPENDIX A

Sampling Procedures

(From EPA Manual SW-846, Third Edition, 1986)

1.0 SAMPLE HANDLING AND PRESERVATION

This section deals separately with volatile and semi-volatile organics. Refer to Section 6.1 and Table 4 of this manual for recommended sample containers, sample preservation, and sample holding times.

2.0 VOLATILE ORGANICS

We use new standard 40 ml glass screw-cap VOA vials with Teflon-faced silicone septum for sampling both liquid and solid matrices. The vials and septum are precleaned by the supplier or manufacturer with certificate according to the EPA procedures.

When collecting the samples, liquids and solids are introduced into the vials gently to reduce agitation which might drive off volatile compounds. Liquid samples should be poured into the vial without introducing any air bubbles within the vial as it is being filled. Should bubbling occur as a result of violent pouring, the sample must be poured out and the vial refilled. Each VOA vial is filled until there is a meniscus over the lip of the vial. The screw-top lid with the septum (Teflon side toward the sample) is then tightened onto the vial. After tightening the lid, the vial is inverted and tapped to check for air bubbles. If there are any air bubbles present the sample must be retaken. Three VOA vials are filled per sample location.

VOA vials for samples with solid or semi-solid (sludges) matrices are completely filled as best as possible. The vials should be tapped slightly as they are filled to try to eliminate as much free air space as possible. Three vials are also filled per sample location.

VOA vials are filled and labeled immediately at the point when the sample is collected. They should **NOT** be filled near a running motor or any type of exhaust system because discharged fumes and vapors may contaminate the samples. The three vials from each sampling location are then sealed in separate plastic bags to prevent cross-contamination between samples particularly if the sampled waste is suspected of containing high levels of volatile organics. (Activated carbon may also be included in the bags to prevent cross-contamination from highly contaminated samples.) VOA samples may also be contaminated by diffusion of volatile organics through the septum during shipment and storage. To monitor possible contamination, a trip blank prepared from distilled deionized water should be carried throughout the sampling, storage, and shipping process.

3.0 <u>SEMI-VOLATILE ORGANICS</u>

(This includes Pesticides and Herbicides)

New precleaned containers (see Section 2.0 Volatile Organics above) used to collect samples for the determination of semi-volatile organic compounds. The sample containers should be of glass or Teflon and have screw-top covers with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. Highly acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may **NOT** be used for the storage of samples due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers are filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g., if an automatic sampler is used), run reagent water through the sampler and use as a field blank.

4.0 SAFETY

Safety should always be the primary consideration in the collection and handling of samples. A thorough understanding of the waste production process, as well as all of the potential hazards making up the waste, should be investigated whenever possible. The site should be visually evaluated just prior to sampling to determine additional safety measures. Minimum protection of gloves and safety glasses should be worn to prevent sample contact with the skin and eyes. As a minimum, a respirator should be worn even when working outdoors if organic vapors are present. More hazardous sampling missions may require the use of supplied air and special clothing. Any sampling program should have the proper safety plan specifying safety procedures and protective equipment, clothing, and monitoring. It is beyond the scope of this manual to design a safety plan for all possible safety requirements for sampling in the field. Field sampling will be performed by the laboratory clients per their safety procedures.

APPENDIX B

Audit Procedures

1.0 PERFORMANCE AUDIT

- 1.1 QA/QC officer submits check sample to the analyst.
- 1.2 Analyst performs test and submits report form according to normal procedures.
- 1.3 QA/QC officer checks laboratory results against the actual concentrations in the check sample.
- 1.4 If any parameter falls outside the acceptance criteria for the method, then the source of the problem must be located, and corrected before further analysis can continue.

2.0 SYSTEMS AUDIT

The systems audit is a qualitative evaluation of the laboratory to ensure that the SOPs are being carried out correctly and that QA/QC procedures are being followed. The QA/QC officer will check the following items:

- 2.1 The analytical instruments must be in proper working order. For the gas chromatograph, there must be a sufficient supply of the proper purity of gases; the proper columns and detectors must be used for each analysis; the detector response should be high enough to meet quantity limits (periodic checks with standards establish the detector response); the gas chromatograph must be in proper working order, and any faults corrected.
- 2.2 The analysts must follow the proper procedures for preparing and analyzing samples, and for reporting results. The QA/QC officer will observe the analytical procedures and check the data analysis and reporting methods. He will note any deviations from established procedures which must be corrected.
- 2.3 The QA/QC officer will check 10% of the laboratory analyses to assure that the data is being reported correctly. This requires checking the chromatograms, verifying peak identifications, and compound quantities.

GLOSSARY

ACCURACY: The degree of agreement of a measurement (or an average of measurements of the same thing) X, with an accepted reference or true value T, usually expressed as the difference between the two values X-T, or the difference as a percentage of the reference or true value 100 (X-T)/T, and sometimes expressed as a ratio X/T. Accuracy is a measure of the bias in a system.

ANALYTICAL BATCH: The basic unit for analytical quality control is the analytical batch. The analytical batch is defined as samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch should be of similar composition.

AUDIT: A systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) performance audits in which <u>quantitative</u> data is independently obtained for comparison with routinely obtained data in a measurement system or, (2) system audits of a <u>qualitative</u> nature that consist of an onsite review of a laboratory's quality assurance system and physical facilities for sampling, calibration, and measurement.

BLANK: A blank is an artificial sample designed to monitor the introduction of artifacts into the process. For aqueous samples, reagent water is used as a blank matrix; however, a universal blank matrix does not exist for solid samples, and therefore, no matrix is used. The blank is taken through the appropriate steps of the process. A <u>reagent blank</u> is an aliquot of analyte-free water or solvent analyzed with the analytical batch. <u>Field blanks</u> are aliquots of analyte-free water or solvents brought to the field in sealed containers and transported back to the laboratory with the sample containers. <u>Trip blanks</u> and <u>equipment blanks</u> are two specific types of field blanks. <u>Trip blanks</u> are not opened in the field. They are a check on sample contamination originating from sample transport, shipping, and from site conditions. <u>Equipment blanks</u> are opened in the field and the contents are poured appropriately over or through the sample collection device, collected in a sample container, and returned to the laboratory as a sample. Equipment blanks are a check on sampling device cleanliness.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check, is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution which is different from the stock used to prepare standards.

CHECK SAMPLE: A blank which has been spiked with the analyte(s) from an independent source in order to monitor the execution of the analytical method is called a check sample. The level of the spike shall be at the regulatory action level when applicable. Otherwise, the spike shall be at five times the estimate of the quantification limit. The matrix used shall be phase matched with the samples and well characterized: for an example, reagent grade water is appropriate for an aqueous sample.

MATRIX SPIKE/DUPLICATE ANALYSIS: In matrix spike/duplicate analysis, predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked, and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at the regulatory standard level or the estimated or actual method quantification limit.

MQL: The method quantification limit (MQL) is the minimum concentration of a substance that can be measured and reported.

PERFORMANCE AUDITS: Procedures used to determine quantitatively the accuracy of the total measurement system or its component parts.

PQL: The practical quantity limit (PQL) is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.

PRECISION: A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Precision may also be described in terms of the relative percent difference (RPD) between two measurements, A and B, defined as RPD = 100 (A-B)/((A+B)/2).

QUALITY ASSURANCE: The total integrated program for assuring the reliability of monitoring and measurement data. A system for integrating the quality planning, quality assessment, and quality improvement efforts to meet user requirements.

QUALITY CONTROL: The routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.

STANDARD CURVE: A standard curve is a curve which plots concentrations of known analyte standard versus the instrument response to the analyte.

SURROGATE: Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction, and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples, and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

WATER: Reagent, analyte-free, or laboratory pure water means distilled or deionized water which is free of contaminants that may interfere with the analytical test in question.



STANDARD OPERATING PROCEDURE

SOIL GAS SAMPLING PROTOCOL

SOPMB01

Revision 1, July 1, 2003

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AMERICAN ANALYTICS SOIL GAS SAMPLING PROTOCOL

1.0 SURVEY DESIGN

- The following is a survey design procedure recommended by the Los Angeles Regional Water Quality Control Board and the DTSC in their Interim Guidance and Advisory for Active Soil Gas Investigation (January 28, 2003). Sample spacing may be modified based on site-specific conditions with Agency approval. When applicable, to optimize detecting and delineating VOCs, the grid spacing should be modified to include biased sampling locations.
- Prepare a scaled facility plot plan indicating potential source areas (underground storage tanks, product dispensers and product lines) and proposed soil gas sample points). The plot plan is to include the location and coordinates of identifiable landmarks such as wells, benchmarks, and street center-lines.
- Create a 20-30 foot grid over the potential source areas and indicate on the grid the soil gas sample points. Locating the sample points at the nodes of the grid may be helpful for establishing a sampling pattern. The size of the grid will depend on the nature of the potential contamination source. If, for example, the potential source is an underground product line, the 20-30 foot grid will extend longitudinally along the line and will become less dense as you move laterally away from the line. For areas on the site in question that are not suspected to be contaminated, establish a maximum of a 100 foot grid to avoid missing potential contamination.
- Initially perform a shallow soil gas survey at approximately 5 feet below grade. Based on the results of this survey, sample to greater depths.
- For areas with known soil contaminants, or where prior soil gas sampling has revealed the
 presence of soil contamination, establish a denser sampling grid (10-20 foot) and sample at
 multiple depths usually in 3 to 5 foot increments. The grid will be designed to adequately
 describe the plume. The number, location and depth of the sampling points will depend on
 the extent of the contamination and the nature of the source.
- Based on real time analytical results provided by the on-site mobile laboratory, field adjustments can be made to the sampling plan regarding the grid density, sample point location and sampling depth. However, field adjustments are acceptable only if the decision-making criteria are included in the work plan and in consultation with the regulatory agency.

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• If certain anomalies are identified with the analytical results for a given sample point, such as numbers 2 to 3 orders of magnitude different from trends indicated by surrounding samples, resample and reanalyze at that point.

2.0 METHODS AND PROCEDURES

2.1 Lithology

Site soil or lithologic information should be used to select appropriate locations and depths for soil gas probes. If on-site lithologic information is not available prior to conducting the soil gas investigation, at least one (1) continuously cored boring to the proposed greatest depth of the soil gas investigation should be installed at the first sampling location, unless specifically waived or deferred by the Agency. Depending on site conditions, additional continuously cored borings may be necessary.

- If low flow or no flow conditions (vacuum readings exceeding approximately 10 inches of mercury or 136 inches of water) are encountered, soil matrix sampling using EPA method 5035A should be conducted in these specific areas.
- If the bottom five (5) feet of a continuously cored boring is composed of clay or soil with a high vacuum reading (see above), the continuously cored boring should be extended an additional five (5) feet to identify soil vapor permeable zones. If the extended boring is also composed entirely of clay, the boring may be terminated. Special consideration should always be given to advancing borings and ensuring that a contaminant pathway is not being created through a low permeability zone.

2.2 Sample Depth

Sample depths should be chosen to minimize the effects of changes in barometric pressure, temperature, or breakthrough from ambient air from the surface; and to ensure that representative samples are collected. Consideration should be given to the types of chemicals of concern and the lithology encountered.

- At each sample location, soil gas probes should be installed at a minimum of one sample depth, generally at five (5) feet below ground surface (bgs).
- Samples should be collected near the lithologic interfaces or based on field instrument readings from soil cuttings and/or cores to determine the location of maximum analyte concentrations at the top or bottom of the interface depending upon the analyte.
- Multi depth sampling is appropriate for any of the following locations:
 - Sites identified with subsurface structures (USTs, sumps, clarifiers, waste or chemical management units), subsurface sources (oil fields, artificial fill, buried animal waste), changes in lithology, and/or contaminated groundwater. Soil gas probes should be emplaced below the base of any subsurface structures, sources or

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- backfilled materials in the vadose zone. Collection of deeper samples should be done in consultation with Agency staff.
- 2. Areas with significantly elevated VOC concentrations detected during shallow or previous vapor sampling.
- 3. Areas where elevated field instrument readings are encountered from soil matrix cuttings, cores or samples.
- 4. In the annular space of groundwater monitoring wells during construction, where assessment of the vertical extent of soil gas contamination is necessary.
- If no lithologic change or contamination is observed, default sampling depths may be selected for multi depth sampling. For example, soil gas samples may be collected at 5, 15, 25, 40 feet bgs until either groundwater is encountered or VOCs are not detected, whichever comes first.
 - 1. Additional samples may be necessary based on site conditions.
 - 2. For preliminary endangerment assessments: When 40 feet bgs is reached, collection of deeper samples may be waived. However, assessment and/or characterization of the deeper vadose zone may be required in the future to protect groundwater resources.

2.3 Sample Collection

Soil gas samples are collected using a soil gas sampling system as shown in Figure 1 in Appendix B. For detailed specifications on the soil gas sampling equipment refer to Table 1 in Appendix A.

The soil gas sampling procedure is performed in three steps: probing, sampling and probe removal. The probe removal step is not performed if the sampling point is to remain for future gas sampling and analysis.

Probing:

- Thread the point holder onto the probe rod and insert the drive tip into the point holder.
- Place the drive cap on the probe rod to protect the threads and drive the rod into the ground using a hydraulic impact direct push technology rig. In areas with limited access use the Concord limited access hydraulic impact push technology rig, an electric impact hammer or slide hammer to drive the rods to depth. Remove the drive cap from the probe rod, thread another segment of probe rod onto the portion of the rod protruding from the ground and drive once again into the ground with the impact hammer. Repeat the same process until the desired depth is reached. Keep

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- track of the probe rod depth by counting the number of three foot segments used during the probing procedure.
- Retract the probe rod by approximately 1" to separate the drive tip from the point holder. This will allow for the soil gas to be drawn through the inner tubing system when a sample is taken.
- Use hydrated bentonite to seal around the drive rod at ground surface to prevent ambient air intrusion from occurring along the point of contact of the rod outer surface with the soil during sampling.

Equilibration Time:

- During probe emplacement, subsurface conditions are disturbed. To allow for subsurface conditions to equilibrate, the probe rod must remain in place for a minimum of 20 minutes prior to commencing sampling.
- Record the probe installation time in the field sampling log book.

Sampling:

- Attach the 0.25" O.D. polyethylene tubing securely to the threaded adaptor and feed the tubing down the inside of the probe rod. When the adaptor hits the point holder, begin to rotate the tubing in a counter clockwise direction in order to thread the adaptor into the point holder. Pull up lightly on the polyethylene tubing to ensure that the threads are engaged and the O-ring forms an airtight seal against the surface of the point holder. Allow 2 feet of tubing to extend past the probe end before cutting. You now have a leak free pathway for the soil gas to travel from the sample point location at depth to the surface.
- Sampling and purging flow rates should not enhance compound partitioning during soil gas sampling. Samples should not be collected if field conditions are as specified in section 2.5 (Field Conditions/Soil Permeability). Obtain the soil gas sample at a volumetric flow between 100 and 200 ml/min to limit stripping, prevent ambient air from diluting the samples, and to reduce the variability of purging rates. The low flow purge rate increases the likelihood that representative samples may be collected. The purge/sample rate may be modified based on conditions encountered in individual soil gas probes. These modified rates, if used, must be documented in the soil gas report.
- Attach the vacuum pump circuit to the line and start the pump with the flow valve initially closed and the isolation valve in the fully open position (refer to Figure 1 in

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Appendix B). Slowly open the flow valve and allow the soil gas to flow through the system for a time period such that the total volume of soil gas pumped approximates the optimum calculated purge volume (refer to Section 2.3 for optimum purge volume determination). Immediately close the isolation valve and allow time for the sample line pressure to return to zero as indicated on the vacuum gauge.

- Record the purge volume, and the evacuation pressure at which the sample is collected in the log book. These numbers will also appear on the final analytical report submitted to the client.
- Proceed to take a sample by penetrating the septum of the sampling tee with a 25 ml gas tight disposable syringe equipped with a luer lock fitting. Slowly retract the plunger until a Twenty (20) ml sample aliquot has been collected, close the luer lock fitting and proceed as quickly as possible to the gas chromatograph for analysis. The actual sample volume injected onto the analytical instrument is ten (10) ml leaving ten (10) ml of the sample in the syringe for re-analysis or other testing as deemed necessary. The sample must be analyzed within thirty (30) minutes from the time of collection. In the event the sample is to be stored prior to injection for a time period greater than five (5) minutes, the syringe must be covered with aluminum foil to prevent degradation of light sensitive compounds. A larger than twenty (20) ml sample aliquot may be taken with a larger capacity disposable syringe to improve sensitivity and achieve lower method reporting limits (MRLs).

Probe Removal:

- After collecting the sample, disconnect the line from the pump train and pull up firmly
 on the line until it releases from the adaptor at the bottom of the hole.
- Remove the line from the probe rod and discard. Decontamination of the
 polyethylene line is not practical due to the low cost of replacement tubing and the
 increased risk of cross contamination if the same line is used over again at another
 sampling point.
- Extract the rods segment by segment from the ground with the direct push technology rig or a probe jack in areas where access is limited. When the final rod equipped with the tip holder is extracted, inspect the O-ring on the line adaptor to ensure that a leak free seal was formed. If the seal is suspect, another sample must be taken.

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2.4 Alternative Sampling techniques

Samples may also be collected in Summa canisters and transported to the fixed laboratory for analysis by EPA methods TO-14 or TO-15. If a Summa canister is used for sample collection, a flow regulator must be placed between the probe and the Summa canister to ensure the Summa canister is filled at the flow rate as specified in section 2.3.

2.5 Optimum Purge Volume Determination

- Before soil gas sampling can be performed, the optimal purge rate and volume must be determined in order to obtain samples that are representative of the volatile organic contaminant levels in the formation around the probe tip. The purge volume or "dead space" volume can be estimated based on a summation of the internal volume of tubing used (4.5 ml/ft), and the volume of annular space around the probe tip (~18 ml). Step purge tests of one (1), three (3), and seven (7) purge volumes are recommended as a means to determine the purge volume to be applied at all sampling points. A typical system "dead" volume when obtaining a five (5) foot soil vapor sample is ~ 50 ml. The procedure of determining the optimum purge volume is by conducting a site specific purge volume versus contaminant concentration test where the VOC levels are expected to be highest. A plot of the contaminant concentration vs purge volume is made (see Figure 4 in Appendix B) and the optimal purge volume is the point at which the contaminant concentration maximizes. The purge time is then set at a value which will generate the optimal purge volume at the specified purge rate.
- The purge test location should be selected as near as possible to the anticipated or confirmed contaminant source, and in an area where soil gas concentrations are expected to be greatest based on lithology. The first purge test location should be selected through the workplan approval process or as a field decision in conjunction with agency staff. If VOCs are not detected for this testing event, a default purge volume of three (3) system "dead" volumes must be used for additional samples taken at the site.
- Additional purge volume tests should be performed to ensure appropriate purge volumes are extracted if:
 - **§** Widely variable or different site soils are encountered
 - § The default purge volume is used and a VOC is newly detected.
- If a new purge volume is selected after additional step purge tests are conducted, the soil gas investigation should be continued as follows.
 - 1. In areas of the same or similar lithologic conditions
 - § Re-sample twenty (20) percent of the previously completed probes. This resampling requirement may be reduced or waived in consultation with agency staff, depending on site conditions. If re-sampling indicates higher

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- detections (e.g.,more than 50 percent difference in samples detected at greater than or equal to 10 ug/L), all other previous probes should be resampled using the new purge volume.
- **§** Continue the soil gas investigation with the newly selected purge volume in the remaining areas.
- 2. In areas of different lithologic conditions: Continue the soil gas investigation with the newly selected purge volume in the remaining areas.
- The purge test data (calculated purge volume, rate and duration of each purge step) should be included in the report to support the purge volume selection.

2.6 Permanent Sample Point Installations

Vapor Sampling Implants (VSI's):

- Permanent sampling implants are installed at sample points where long term monitoring is required. An example of an application of this nature is long term sampling implant installation to evaluate the effectiveness of vapor extraction systems (VES) (refer to Figure 5 in Appendix B for a representation of a soil gas sampling implant).
- The implants are constructed of stainless steel wire screen with an adaptor on the end to connect the polyethylene sampling line.
- The installation involves driving the probe rods to the desired depth and lowering the implant and sampling line down the inside of the probe rod to depth. Sand is then poured down the inside of the probe rod followed by bentonite to seal the implant in place.

The implant should be emplaced midway within a minimum of one (1) foot of sand pack. The sand pack should be appropriately sized (no smaller than the soil granules comprising the adjacent formation) and installed to minimize disruption of flow to the implant. At least one (1) foot of dry granular bentonite should be emplaced on top of each sand pack to preclude the infiltration of hydrated bentonite grout. The probe rods are then removed with the hydraulic impact push technology rig or the jack puller and the remaining hole is backfilled with hydrated bentonite grout. The end of the sampling line is marked appropriately and fitted with an adaptor for easy connection of the pumping circuit when a sample is taken.

Nested Vapor Sampling Implants:

- Nested soil gas sampling implants are used for long term monitoring of VOC soil gas concentrations at multiple depths (refer to Figure 6 in Appendix B for a representation of a nested soil gas sampling implants).
- The implants are installed as described in the previous section and each sampling point at a given depth is isolated from the other by a hydrated bentonite grout plug.
- For deep nested probe installations the use of a downhole probe support may be required. Such support may be constructed from a one (1) inch diameter bentonite/cement grouted PVC pipe or other solid rod.

2.7 Sampling Problems and Troubleshooting

Zone of Influence, Atmospheric Breakthrough, Leak Test:

Depending on the specific lithologic unit in which the sampling is taking place, the pumping process prior to sampling will effect a larger or smaller volume of space surrounding the sample probe tip. The effected volume of space is referred to as the zone of influence. This is the zone from which soil gas can migrate to the probe tip during the pumping process. The zone of influence is a function of lithology, land cover, drive point construction and sample purge time/rate/volume. For ex. when pumping the same volume, the zone of influence will be much greater for a soil with a small effective porous space as compared to that of a soil with a larger effective porous space. When sampling soil gas, care must be taken so that the vertical zone of influence does not intersect the ground surface. If this occurs, atmospheric air will be drawn through the ground to the sample probe tip and dilute the soil gas sample, producing artificially low results for the VOC's. This problem usually occurs with shallow soil gas sampling (5 feet) but can occur when sampling at larger depths. With knowledge of the lithology, the purge rate/time/volume can be adjusted so that the zone of influence does not intersect the ground surface. In certain soil types the outer surface of the soil gas sample probe rod may not form a good seal with the soil formation. If this occurs atmospheric breakthrough may occur along the outer surface of the probe rod down to the sample probe tip and dilute the soil gas sample, producing artificially low results for the VOC's. Atmospheric breakthrough along the outer surface of the rod in contact with the soil can be determined by performing a leak test using tracer compounds such as pentane, isopropanol, isobutene, propane, and butane if a detection limit of 10ug/L or less can be achieved for the compound selected. Some of these compounds are present in commercially available products such as shaving cream. The shaving cream is applied at the

ground surface where the soil gas probe rod contacts the ground and at the top of the rod in the annular space between the inner surface of the rod and the polyethylene tubing. This procedure must be performed at each and every sample location. If atmospheric breakthrough is occurring, the tracer compounds present in the shaving cream will travel with the atmospheric air to the sample point location and will ultimately arrive in the gas tight syringe during the sampling event. The detection of the tracer compounds from the analysis of the soil gas confirms the occurrence of atmospheric breakthrough. To prevent atmospheric breakthrough, the sample rod is sealed at the point of contact with the surface with hydrated bentonite.

The leak test must include an analysis of the leak check compound. If a leak check compound is detected in the sample the following actions must be taken.

- § The cause of the leak should be evaluated, determined and corrected through confirmation sampling.
- § If the leak check compound is suspected or detected as a site specific contaminant, a new leak check compound must be used.
- § If a leak is confirmed and the problem cannot be corrected, the soil gas probe should be properly decommissioned.
- § A replacement probe should be installed at least five (5) feet from the original probe decommissioned due to confirmed leakage, or consult with the agency.
- § The leak check compound concentration detected in the soil gas sample should be included and discussed in the report.

Field Conditions/Soil Permeability:

- Soils that are saturated with water, or soils that are comprised of tightly packed fine
 particulates resulting in a very small effective porous space, may show no or very
 low permeability to soil gas regardless of the vaccum applied to the sampling
 system.
- When a sampling probe is inserted in a soil exhibiting the above properties, the vacuum gauge will not return to zero when the vacuum pump is turned off and the isolation valve is closed. If this problem is universally present throughout the site the soil vapor sampling must cease. In addition it is recommended by the agency that soil vapor sampling should not be conducted during or immediately after a significant rain event (0.5" or greater) or onsite watering.
- In some soils exhibiting low permeation to soil vapor (no or low flow conditions) the
 needle on the vacuum gauge may return to zero very slowly. The time it takes the
 needle to return to zero is called the recovery time and is indicative of relative soil

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permeability. Recovery times that are greater than 10 minutes should be considered suspect and if the recovery time exceeds 10 minutes the sampling probe should be removed and a sample taken at a different location where the soil may exhibit a greater permeability to soil gas.

System Leaks:

• When the vacuum pump is turned off, and the isolation valve is closed, the needle on the vacuum gauge should gradually return to zero upon pressure equilibration. If the needle returns to zero rapidly, this may indicate the presence of a leak in the sampling train before the isolation valve. Leaks in the system result in artificially low results for the VOC's and sampling must be discontinued until the leak is found and the problem corrected. Leaks can be found by isolating segments of the system and applying a vacuum to each segment to see if it retains the vacuum. Prior to sampling, it is advisable to plug the end of the sampling train and apply a vacuum to it to check for leaks.

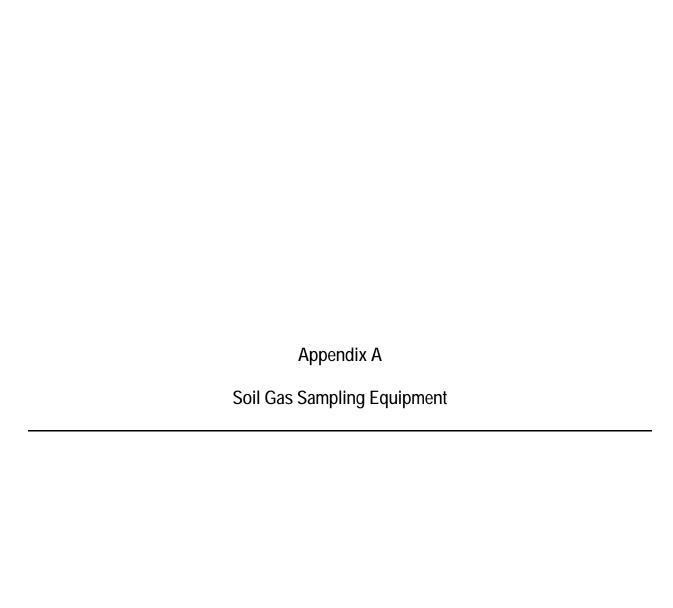
Decontamination:

- In order to avoid cross-contamination problems, the soil vapor drive tip holders, the
 adaptors that connect the polyethylene tubing to the tip holder and the drive rod to
 which the tip holder is attached are decontaminated in the field. The process
 includes soap and water cleaning with a phosphate free detergent followed by a two
 stage rinsing with organic free water and allowing to air dry.
- The polyethylene sampling line is discarded after each sampling event to avoid cross contamination problems.

2.8 Sample Analysis

Soil gas samples are analyzed by injection of the sample through a purge and trap system onto a Gas Chromatograph equipped with a mass spectrometer detector capable of detecting the parameters of interest with the necessary sensitivity. Purge and trap systems used include: OI analytical model 4560, and Tekmar model LSC 2000. Gas chromatographs include: Hewlett Packard Model 5890 Series II. Detectors include: Hewlett Packard Model 5971 Mass Spectrometer. For a detailed description of the analytical procedures used for soil gas analysis refer to the American Analytics Standard Operating Procedure (SOP) GC006.

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APPENDIX A

Table 1 Soil Gas Sampling Equipment List

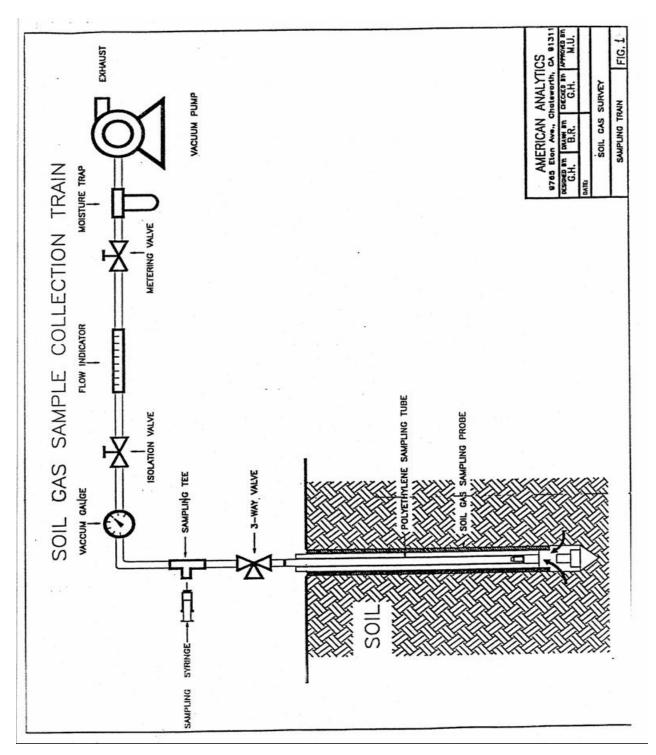
Item #	Item Description	Usage	Manufacturer
	Sam	pling Equipment	
1	Geoprobe model 5400 Truck Mounted Direct Push Technology Sampling Rig	Drive probe rods into the subsurface.	Geoprobe
2	Concord Limited Access Direct Push Technology Sampling Rig	Drive probe rods into the subsurface in areas where access is limited.	Concord
3	Machined steel expendable drive point.	Soil penetration.	Geoprobe
4	Expendable point holder.	Holds drive point, threaded to accept sample line with adaptor.	Geoprobe
5	Sample line adaptor.	Connects sample line to point holder.	Geoprobe
6	Probe rods (carbon steel, 36" length, 1" O.D. x 0.5" I.D.)	Probe to desired sample depth by mechanical impact.	Geoprobe
7	Sample line (Polyethylene, 0.25" O.D., 0.17" I.D., 4.46 ml/ft Internal Vol.)	Provides soil gas path from sample point at desired depth to surface.	Geoprobe
8	Manual probe rod drivers. (Electric Hydraulic Hammer or Manual Impacter).	Drive probe rods into the ground to desired sampling depth.	Geoprobe
9	Probe rod jack.	Removes probe rods when there is limited access for the push technology hydraulic hammer rig.	Geoprobe
10	Sampling tee (1/4" Stainless Steel).	Take soil gas sample with gas tight syringe.	Swagelok
11	Isolation Valve.(2-way ball valve)	Isolate sampling train prior to sampling.	Swagelok
12	3-way Valve. (Ball)	Purge system with inert gas after sampling.	Swagelok
13	Metering valve. (Union Bonnet)	Meter flow of soil gas through the sampling train.	Swagelok
14	Vacuum gauge.(30 to 0"Hg)	Take sampling train vacuum readings.	Weksler Instruments
15	Flow meter (40-200 ml/min flow range) with bar graph display.	Take soil gas flow readings through the sampling train.	

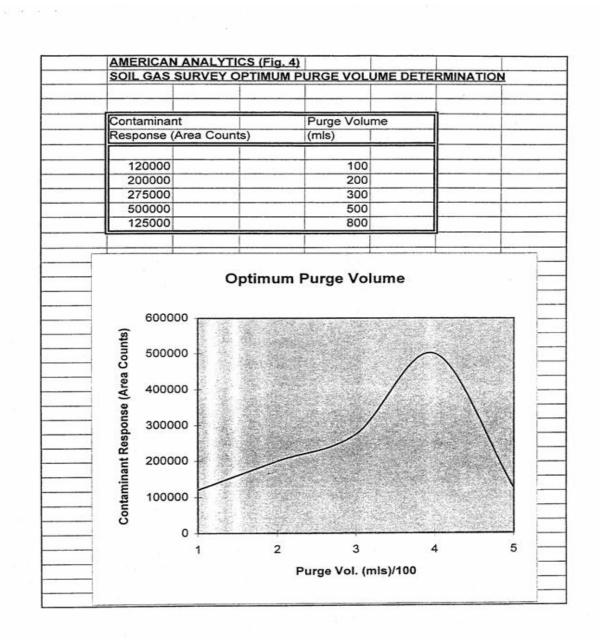
Table 1 (Continued) Soil Gas Sampling Equipment List

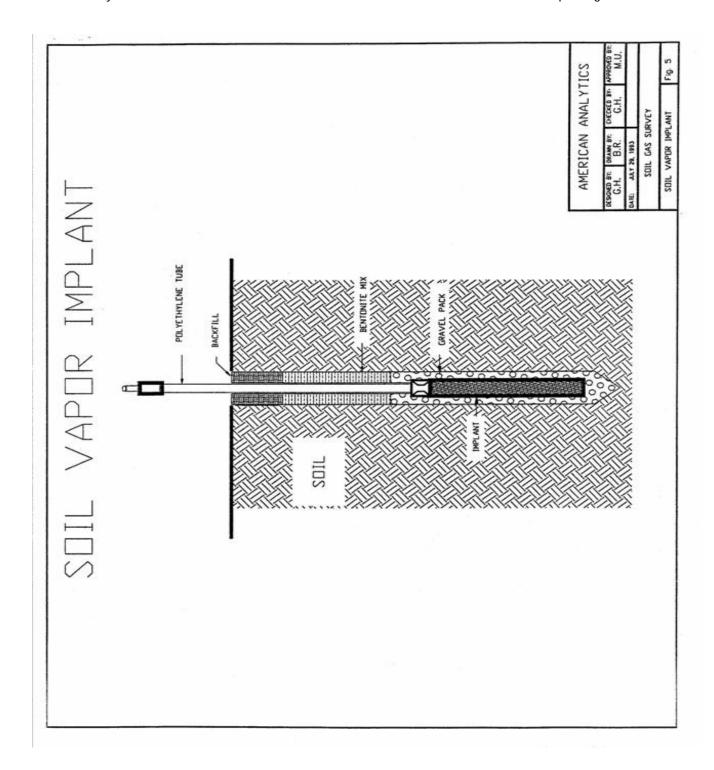
Item #	Item Description	Usage	Manufacturer
16	Vacuum Pump (0.5 cfm @ 18" Hg diff. press.)	Apply vacuum to sampling train to extract soil gas from the ground.	Cole Parmer
17	Disposable Gas Tight Syringe	Take soil gas sample for injection into the gas chromatograph.	Various
	Sa	ample Analysis	
1	Gas Chromatograph (HP model 5890 series II equipped with 60 m, J&W DB-VRX volatiles column).	Compound separation	Hewlett-Packard
2	Mass Spectrometer (HP model 5971)	VOCs Detection	Hewlett-Packard
3	FID Detector (OI model 4430)	Methane Detection	OI Corp.
5	Purge and Trap (Tekmar LSC 200)	Sample concentration and introduction into the gas chromatograph	Tekmar
6	Purge and Trap (OI model 4560)	Sample concentration and introduction into the gas chromatograph.	OI Corp.

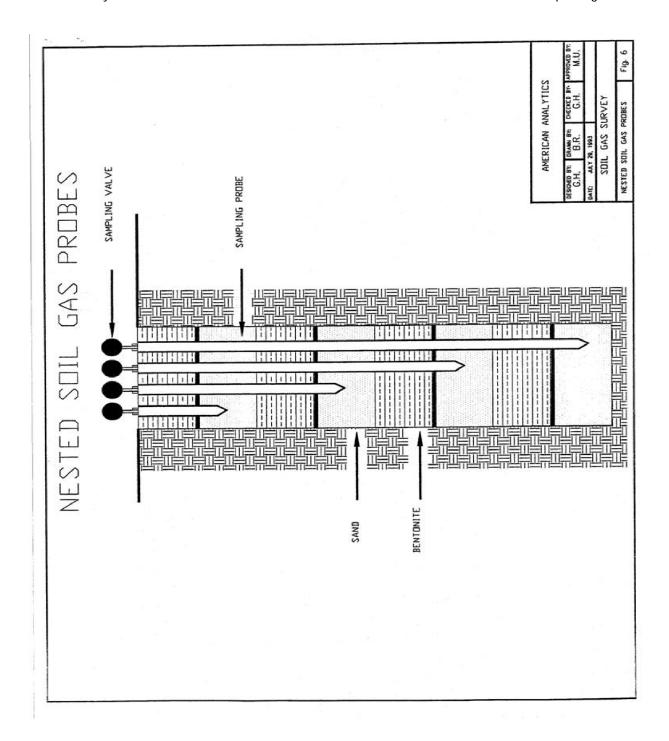
Appendix B

Figures and Plots









STANDARD OPERATING PROCEDURE

METHOD FOR DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN SOIL GAS BY GC/MS DTSC

SOPMB02

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American Analytics SOP: MS005

METHOD FOR DETERMINATION OF VOLATILE ORGANICS IN SOIL GAS

1. <u>SUMMARY OF METHOD</u>

This method is used to determine the concentration of volatile organic compounds in soil gas using a gas chromatograph equipped with a Mass Spectrometer. While in the purge mode, an aliquot of the soil gas sample is introduced by use of a gastight syringe through the purge vessel of a purge and trap unit onto the trap. The trap is rapidly heated and the volatile compounds are carried into a gas chromatograph equipped with a capillary column for compound separation, and a MSD for compound detection. The gas chromatograph is programmed to go through a temperature program during which all compounds of interest are eluted into the detector system.

For specific projects where greater sensitivity is required, a larger volume of the sample may be injected in order to achieve lower detection limits.

The following is the target analyte list and their associated MRLs for the soil gas analysis:

Primary Target Compounds	MRL (ug/L)
Carbon tetrachloride	1
Chloroethane	1
Chloroform	1
1,1-Dichloroethane	1
1,2-Dichloroethane	1
1,1-Dichloroethene	1
cis-1,2-Dichloroethene	1
trans-1,2-Dichloroethene	1
Methylene chloride	1
Tetrachloroethene	1
1,1,1,2-Tetrachloroethane	1
1,1,2,2-Tetrachloroethane	1
1,1,1-Trichloroethane	1
1,1,2-Trichloroethane	1

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Primary Target Compounds	MRL (ug/L)
Trichloroethene	1
Vinyl chloride	1
Benzene	1
Toluene	1
Ethylbenzene	1
m,p-Xylenes	1
o-Xylene	1
Trichlorofluoromethane (Freon 11)	1
Dichlorodifluoromethane (Freon 12)	1
1,1,2-Trichloro-trifluoroethane (Freon 113)	1

Other Target Compounds	
Ethyl tert-Butyl Ether (ETBE)	1
Tert-Amyl Methyl Ether (TAME)	1
Methyl tert-Butyl Ether (MTBE)	1
Tert-Butanol (TBA)	1
Methyl ethyl ketone (MEK)	1
Meythyl isobutyl ketone (MIBK)	1
Ethylene dibromide (EDB)	1

Tracer Compounds	
Isobutane	10
Propane	10

Other specific site contaminants including all of the routinely analyzed compounds by 8260B may be analyzed by this method. The tracer compounds are found in shaving foams. The shaving foam is Babasol brand and can be purchased from any grocery stores. The tracer compounds need to be calibrated and monitored by the chemist and the result needs to be included in the analytical report.

2. <u>INTERFERENCES</u>

The analysis of a highly contaminated sample may result in carryover of the contaminants to the next sample being analyzed. To avoid this problem, a blank sample should be analyzed after the highly contaminated sample to check for cross contamination problems. Cross contamination problems can also occur by using contaminated syringes that have not been purged properly after being used for transfer of a contaminated sample. The trap and other parts of the purge and trap system are also subject to contamination and may require a series of bakeouts at elevated temperatures to eliminate the problem.

3. <u>SAFETY</u>

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; each chemical should be treated as a potential health hazard and exposure to these chemicals should be reduced to the lowest possible level by whatever means available.

- X Safety glasses shall be worn at all times when working in the laboratory.
- X Gloves shall be worn when handling samples or standards.
- X A respirator should be worn when preparing standards.
- X Care should be taken when handling syringes. Syringes are sharp objects that can easily penetrate the skin and introduce a toxic substance into the body.

All standard and solvent containers shall be clearly labeled to indicate their contents.

Methanol is a solvent that is frequently used for this test. Methanol can cause blindness if ingested, so care should be used when handling this solvent. Methanol is also extremely flammable and shall be stored in the flammable storage cabinet.

Information on the hazards associated with chemicals that the chemists could be exposed to while performing this test is available in the stationary laboratory MSDS files.

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4. APPARATUS AND MATERIALS

4.1 Gas Chromatograph

- 4.1.1 <u>Gas Chromatograph</u>: Hewlett Packard model 5890 with multiple ramp temperature programming capabilities.
- 4.1.2 <u>Column</u>: J&W DB-VRX, 60m, 0.25 mm ID, 0.25 micron film thickness, 260 °C upper temperature limit. An equivalent column may be used if the J&W column is not available.
- 4.1.3 Detectors: HP 5971 MSD capable of scanning 35-300 amu.
- **4.2 Syringes**: Gas-tight syringes equipped with a luer lock valve capable of dispensing 1, 5 and 25 ml of vapor. If lower detection limits are required, syringes capable of dispensing larger vapor volumes can be used. Disposable polyethylene syringes may be used when applicable.
- **4.3 Microsyringes**: A series of Hamilton microsyringes having dispensing capacities of 10, 25, 50, 100, 500 and 1000 ul. The microsyringes are used for standard preparations.
- **4.4 Balance**: Sartorius Analytical Balance capable of weighing to the nearest 0.0001 gr. The balance is used when required to weigh neat standards for the preparation of stock standard solutions.
- **4.5 Tedlar Bags:** 1L and 5L Tedlar Bags provided by SKC Inc. The Tedlar bags are used for:
 - X Storing ultra high purity nitrogen for blank runs and syringe purging between sample runs.
 - X Dilution of samples containing target analytes which exceed the linear range of the calibration curve.
 - X Storing samples which require confirmation runs by GC/MS in the stationary laboratory.
- **4.6 Disposable Pipets**: Pasteur.

4.7 Purge and Trap Devices: TEKMAR LCS 2000 equipped with a 16 position auto-sampler. The trap used is VOCARB 3000.

4.8 Data System: Processing of the chromatographic data and report generation is performed by the HP CHEMSTATION equipped with Enviroquant for data acquisition and processing.

5. <u>REAGENTS</u>

- **5.1 Reagent Water**: Purchased distilled water which has been purged with nitrogen for a time period of no less than one hour prior to use.
- **5.2 Methanol**: Purge & Trap grade supplied from Baxter or from EM-Science.

5.3 Gases

5.3.1 <u>Helium</u>: Grade 5 with a purity of 99.999% is used as a carrier gas for the GC column.

5.4 Stock Standards

5.4.1 <u>Initial Calibration and Daily Calibration Standards</u>: The standards are purchased from Accustandard at concentrations of 2000 ug/l. The following is the list of the stock standard mix solutions which are used to prepare the working solution standard mixes.

STANDARD MIX COMPOUND LIST	CONCENTRATION (ug/ml)
VOC MIX (23)	2000
Oxygenates Mix (5)	2000

- 5.4.2 <u>Tuning standard</u>: The tuning compound (bromofluorobenzene) stock solution is purchased from Accustandard at a concentration of 2000 ug/ml.
- 5.4.3 <u>Internal Standard and Surrogate Mix</u>: The stock standards are purchased from accustandard. The following is the list of the

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concentrations and the compounds used to prepare the internal standard and surrogate working solution mix:

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INTERNAL AND SURROGATE STANDARDS	CONCENTRATION (ug/ml)
Pentafluorobenzene (IS1)	2000
Chlorobenzene-d5 (IS2)	2000
1,4-Dichlorobenzene-d4 (IS3)	2000
Dibromofluoromethane (SS1)	2000
Difluorobenzene (SS2)	2000
Toluene-d8 (SS3)	2000

5.4.4 <u>Laboratory Control Check Sample (LCCS)</u>: The laboratory control check sample mix is purchased from Accustandard at a concentration of 200 ug/ml. The following table lists the components contained in the LCS mix.

STOCK LCS MIX COMPONENTS					
Benzene	Vinyl chloride				
Cis-1,2-Dichloroethene	1,1,2,2-Tetrachloroethane				
Carbon tetrachloride	Tetrachloroethene				
Chlorobenzene	Trichloroethene				
Chloroethane	1,1,1-Trichloroethane				
Chloroform	Methylene chloride				
Chloromethane	1,1-Dichloroethane				
1,2-Dichloropropane	1,1,2-Trichloroethane				
1,1-Dichloroethane	m,p-Xylenes				
1,2-Dichloroethane	o-Xylene				
Ethylbenzene					
Toluene					
1,1-Dichloroethene					
trans-1,2-Dichloroethene					

5.4.5 Working Solutions (Preparation)

5.4.5.1 Calibration and Daily Calibration Check Standard Mix:
Prepare by diluting each of the stock solution mixes in 10 ml of methanol. Store the working solution in a teflon sealed mininert screw-cap vial and maintain refrigerated at 4 deg C. Replace the working solution every six months and dispose of appropriately. The following is the dilution table for preparing a 20 ug/ml calibration standard:

STOCK MIX	CONCENTRATION (ug/ml)	VOLUME OF STOCK MIX USED (ml)	FINAL CONCENTRATION (ug/ml)
VOC MIX (23)	2000	100	20
Oxygenates Mix (5)	2000	100	20

- 5.4.5.2 Internal Standard and Surrogate Standard Mix: Prepare by diluting 20 ug/ml of the Internal Standard / Surrogate stock solutions in 10 ml of methanol to prepare a working solution at a concentration of 50 ug/ml. Store the working solution in a teflon sealed Mininert screw-cap vial and maintain refrigerated at 4 °C. Replace the working solution every six months and dispose of appropriately.
- 5.4.5.3 <u>Laboratory Control Check Sample (LCCS)</u>: Prepare by diluting a 400 ul aliquot of the volatile compound mix in 10 ml of methanol to produce a working solution at a concentration of 20 ug/ml. Store the working solution in a teflon sealed Mininert screw-cap vial and maintain refrigerated at 4 °C. Replace the working solution every six months and dispose of appropriately.

6. PROCEDURE

6.1 Gas Chromatography Conditions: Set initial column temperature to 40 °C for 5 min and ramp at 10 °C/min to 225 °C and hold for 0 min, then ramp at 20 °C/min to 250 °C and hold for 3.25 min. The total run time is 28 min.

To increase sample throughput, an alternative temperature program with shorter run time can be used. The instrument is calibrated for this temperature program however, due to the increased temperature ramp rate, two of the analytes co-elute and if identified in the sample the original temperature program must be used to properly identify and quantify these analytes. The shorter temperature program is as follows: Set initial column temperature to 40 °C for 2 min and ramp at 12 °C/min to 148 °C and hold for 0 min, then ramp at 26 °C/min to 226 °C and hold for 0 min for a total run time of 19 min.

- 6.2 Purge and Trap Conditions: Set the sample purge time to 6 min and the trap dry purge time to 3 min. A shorter dry purge time can be used if it is determined that the amount of water desorbed from the trap does not cause any interference. Set the desorb preheat temperature at 220 °C and the desorb temperature at 240 °C for 2 min. Set the trap bake temperature at 270 °C for 6 min. Set the temperature of the transfer line and the valve to 110 °C to avoid condensation of the organics on the inner surfaces of these components.
- **6.3 BFB Tune Criteria:** Demonstrate that the GC/MS meets the ion abundance criteria by analyzing 50 ng of BFB initially and every 12 hours thereafter. The BFB ion abundance criteria are as follows Tuning:

MASS	ION ABUNDANCE
50	15 to 40% of mass 95
75	30 to 60% of mass 95
95	Base peak,100% of mass 95
96	5 to 9% of mass 95
173	Less than 2% of mass 174
174	Greater than 50% of mass 95

MASS	ION ABUNDANCE
175	5 to 9% of mass 174
176	95 to 101% of mass 174
177	5 to 9% of mass 176

6.4 Instrument Calibration

Volatile Organics: The instrument is initially calibrated for all target analytes listed in the table in Section 1 of this procedure. The instrument calibration procedure is as follows. Prepare six calibration standards at levels of 0.5, 2, 10, 20, 50, and 100 ug/L by injecting 0.25, 1, 5, 10, 25, and 50 ul of the VOC calibration standard working solution respectively into 10 ml of purged water placed in a 25 ml gas-tight syringe equipped with a luer lock valve. The concentration of the low level standard must not exceed three times the Method Detection Limit (MDL). Add 5 ul of the internal standard and surrogate working solution to each of the five calibration standards and begin to analyze. When the analysis of the five standards has been completed, update the calibration ID file. software will calculate the response factor of the analytes for each of the five calibration levels using the internal standard method. The software also calculates the average response factor, which is subsequently used to calculate the concentrations of unknowns in the samples, and the percent Relative Standard Deviation (%RSD) which must be below 20% for all analytes.

- 6.5 Instrument Blank: Prior to the analysis of samples, bake the trap on the purge and trap unit for 12 min and bake the gas chromatograph column at 250 °C for the same time period. This will eliminate contaminants that may have accumulated on the system from the previous run. Measure a 10 ml aliquot of reagent water in a 25 ml gas-tight syringe, inject 5 ul of the internal standard and surrogate working solution through the valve into the syringe, introduce into the purge vessel and begin purging. Following the analysis, if the instrument blank is found to be contaminant free proceed with the analysis of the continuing calibration standard.
- 6.6 Daily Calibration Check Standard: Analyze a Daily Calibration Check Standard to verify the validity of the calibration curve. Inject 10 ul of the VOC working solution through the valve of a 25 ml gas-tight syringe containing 10 ml of reagent water, inject 5 ul of the internal standard and

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surrogate working solution into the syringe, transfer the standard to a purge vessel and analyze.

- 6.7 Method Blank: Vapor samples to be analyzed must be preceded by a method blank to determine the presence of background contamination. Measure a 10 ml aliquot of reagent water in a 25 ml gas-tight syringe, inject 5 ul of the internal standard and surrogate working solution through the valve into the syringe, introduce the contents of the syringe into the purge vessel and begin purging. At some point into the 2 min. purge cycle introduce slowly into the purge vessel 10 ml of nitrogen gas by use of a 25 ml gas-tight syringe. The nitrogen gas is carried onto the trap by the purge gas along with the internal standard and surrogate compounds and subsequently desorbed into the gas chromatograph for analysis. A Method Blank is analyzed at the beginning of each day and when necessary during the course of the day depending on the extent of contamination of the samples.
- **6.8 Equipment Blank**: To ensure that the sampling train is contaminant free, prior to taking a sample, ambient air is drawn through the system a sample of which is analyzed to determine the presence of target analytes. If the ambient air is suspected to be contaminated, a contaminant free source must be used such as a nitrogen or air cylinder of known purity. The sample is taken with a gas-tight syringe and analyzed the same as the method blank.
- 6.9 Laboratory Control Check Sample: A minimum of two QC check samples must be analyzed each working day one at the beginning and one at the end of the day thus bracketing the analysis of the environmental samples. Measure a 10 ml aliquot of reagent water in a 25 ml gas-tight syringe, inject 5 ul of the internal standard and surrogate working solution through the valve into the syringe and 5 ul of the LCS working solution. Introduce the contents of the syringe into the purge vessel and begin analysis.
- 6.10 Samples: With a gas-tight syringe, penetrate the sample tee septum on the sampling train and slowly take a soil gas sample for analysis. (For a detailed description of soil gas sampling procedures refer to the American Analytics sampling protocol). Measure a 10 ml aliquot of organic free water in a 25 ml gas-tight syringe, inject 5 ul of the internal standard and surrogate working solution through the valve into the syringe, introduce the contents of the syringe into the purge vessel and begin purging. At

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> some point into the 2 min. purge cycle introduce slowly into the purge vessel the 10 ml aliquot of the sample. The sample is carried onto the trap by the purge gas along with the internal standard and surrogate compounds and subsequently desorbed into the gas chromatograph for analysis. The sample must be submitted to the mobile laboratory as quickly as possible (within 30 min) to preserve the integrity of the sample. If target analytes are present at concentrations above 50% of the highest standard in the calibration curve, the sample must be reanalyzed at a dilution. This is accomplished by injecting a smaller aliquot of the sample or performing a dilution on the sample in a known volume of nitrogen gas contained in a Tedlar bag. The concentration of the analytes in the diluted sample should lie within the mid to upper half of the calibration curve. In order to achieve lower detection levels, the analyst may choose to inject 100 ml of the vapor sample.

6.11 Sample Duplicates: Sample duplicates are analyzed with every analytical batch to obtain precision data about the sampling and analytical methods. The duplicate samples are analyzed in the same manner as the samples as described in Section 6.9.

7. **QUALITY CONTROL**

- 7.1 Analytical Batch: The samples to be analyzed, the instrument, method and equipment blanks, the tune, the daily calibration check standard, sample duplicates and the Laboratory Control Check Samples analyzed within a 12 hr period, comprise a group which is defined as the analytical batch.
- 7.2 **Initial Calibration**: Initial calibration is performed for all compounds listed in the table appearing in Section 1 of this SOP. The calibration is performed by analyzing standards at six concentrations of 0.5, 2, 10, and 100 ug/L in 10 ml of organic free water. If under time constraints in the field, the calibration can be performed using a minimum of three concentrations however, in both cases the low level standard must not be higher than three times the method detection limit. RSD of the response factors for each compound at each different level must not exceed 20% for all target analytes (except for Freons 11,12, and 113, chloroethane and vinyl chloride that must be 30%) or the calibration Identification and quantitation of target must be performed again. analytes must be based on calibration under the same conditions (i.e.

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column, detector, and temperature program etc.). For the same reason if the shorter temperature program is used, the instrument must be calibrated under the same conditions or quantitation of the target analytes will not be possible.

- 7.3 Instrument/Method/Equipment Blanks: The concentration of target analytes in the Instrument/Method/Equipment Blanks must be below the method reporting limit (MRL) for any specific compound. If the blanks do not meet these criteria, they must be reanalyzed. If the problem reoccurs, the source of the contamination must be identified and the problem corrected before samples can be analyzed.
- 7.4 Daily Calibration Check Standard: A midpoint calibration standard, including every compound expected or detected at the site is analyzed prior to the analysis of any samples. The response factor for all of the target compounds (except for freons 11,12,113, chloroethane and vinyl chloride) must be within 15% of the average RF of the initial calibration curve RF. The RF for freons 11,12,and 113,chloroethane, and vinyl chloride must be within 25%. In case that any of the target compounds fail the above criteria and the response is on the high end, the analyst may continue with the analysis. If the failed compound is not detected in any of the samples, then the data is valid and does not need to be qualified. Conversely, if the analyte is detected, then the result is biased high and must be qualified as estimated. If the response of the failed analyte is on the low end, then the analyst must demonstrate that the instrument is capable of detecting the analyte at the MRL by analyzing a standard at the MRL. If the analyte is detected at the MRL, then the analysis may continue and all detected data must be qualified as estimated for this compound. If the analyte is not detected at the MRL, then the analyst must stop the analysis and recalibrate the instrument.
- 7.5 Laboratory Control Check Samples: A Laboratory Control Check Samples obtained from a source different from the calibration standards or standard mix with a different lot number must be analyzed at the end of the batch each working day. The target compounds must be checked and the RF must meet the same criteria as the daily CCV in section 7.4.
- **7.6 Surrogates**: The percent recovery for all surrogates must be within 75 to 125. If the surrogate recovery for a sample is out of control, the sample

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must be reanalyzed to verify that the out of control recovery is not due to a problem with the analytical equipment such as a leak in the system.

7.7 Internal Standards: The internal standard area counts in any sample must fall between 50 and 200% of the average internal standard area from the initial calibration runs. If an out of control situation occurs, verify if the problem is isolated to one sample or if it occurred throughout the entire run. An isolated incident could possibly be attributed to a localized leak in the purge tube whereas a general occurrence requires that the instrument may require troubleshooting and recalibration in order to correct the problem.

7.8 Sample Duplicates: The results obtained from the analysis of the sample and the duplicate sample must show good agreement as indicated by the Relative Percent Difference (RPD) which should be less than 30. If the RPD is greater than thirty this implies the possibility of a problem with the sampling procedure or the analytical method and corrective action must be taken after the source of the problem is identified.

8. CALCULATIONS DATA REVIEW AND CONFIRMATION

8.1 Calculations: Use the internal standard method of quantitation. The data system automatically calculates the amount of the analyte in ug/L by using the calibration information stored in the method (average response factors for each analyte), the area of the quant ion for the targeted analyte and the area of the internal standard from the analytical run. The name of the targeted analyte, quant ion, retention time, and the amount in the appropriate units are displayed on the analytical report which is generated by the data system. If manual calculations are necessary, the following formula can be used for calculating the amounts of targeted analytes in samples.

 $Amt_c = A_c/A_{is}*Amt_{is}*RF*DF$

Where: A_c is the area of the component, A_{is} is the area of the internal standard, Amt_c is the amount of the component in ug/L, Amt_{is} is the amount of the internal standard in ug/L, RF is the response factor from the calibration data and DF is the dilution factor.

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8.2 Data Review: An analyte is positively identified only if the elution time of the analyte falls within the elution window and the ion spectra of the analyte matches the one of the reference standard and the primary and the secondary ion are present at the right ratio. The sample chromatogram must be inspected for the presence of the tracer compound (Isobutene). If the tracer compound is detected in any of the samples, the sampling person must be informed immediately ,the source of the problem must be identified, and the sample must be recollected and reanalyzed. For sampling collection refer to the AA sampling SOP.

9. REPORTING OF SAMPLE RESULTS AND QA/QC INFORMATION

- X Provide the date, time of injection and analytical conditions for all environmental and QA/QC samples.
- X Report all concentrations in ug/L
- X Report for the most recent initial calibration, the retention time and the average response for each compound.
- X Tabulate and report for the Laboratory check samples, the true concentration, detected concentration and percent difference for each compound.
- X Tabulate and report for all environmental samples including duplicates, the sample identification, sampling depth, purge volume, vacuum pressure, sampling time, injection time, injection volume, results and any other sampling or analytical remarks.
- X Raw data such as chromatograms for calibration standards, Laboratory check samples and environmental samples are submitted upon request.
- X Sample report forms containing all required sampling, analytical and QA/QC information are attached for references.

10. REFERENCES

USEPA 8260B, "SW-846 Test Methods for Evaluating Solid Waste", Third Edition.

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The Interim Guidance for Active Soil Gas Investigation (March 14, 1996)

Advisory, Active Soil Gas Investigation. January 28, 2003

		Reporting	Surrogate	Duplicate	Matri	x Spike	Blank Spi	ke / LCS
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD

Carbon Chain Characterization 8015M in Water (EPA 8015M)

Preservation:Cool 4°C							
Container:02_1000mL Amb	er Glass	Amou	ınt Required:1 Liter	H	old Time:	14 days	
Cool to 4° C							
C6-C8	0.010	0.010 mg/L					
C8-C10	0.010	0.010 mg/L					
C10-C12	0.010	0.010 mg/L					
C12-C14	0.010	0.010 mg/L					
C14-C16	0.010	0.010 mg/L					
C16-C18	0.010	0.010 mg/L					
C18-C20	0.010	0.010 mg/L					
C20-C22	0.010	0.010 mg/L					
C22-C24	0.010	0.010 mg/L					
C24-C26	0.010	0.010 mg/L					
C26-C28	0.010	0.010 mg/L					
C28-C32	0.010	0.010 mg/L					
C32-C34	0.010	0.010 mg/L					
C34-C36	0.010	0.010 mg/L					
C36-C40	0.010	0.010 mg/L					
C40-C44	0.010	0.010 mg/L					
TPH (C6-C44)	0.10	0.10 mg/L					
surr: o-Terphenyl			50 - 150				
Diesel Range Organics as Diesel	0.10	0.10 mg/L		70 - 130	30	75 - 125	30

		Reporting	Surrogate	Duplicate	Matri	x Spike	Blank Spik	e / LCS
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
Carbon Chain Characterizatio Preservation:Cool 4°C	n 8015M in So	il (EPA 8015	M)					
Container: 10_Metal Sleeve	e Cool to 4° C	Amo	unt Required:	250 grams	I	Hold Time:	14 days	
C6-C8	1.0	1.0 mg/kg						
C8-C10	1.0	1.0 mg/kg						
C10-C12	1.0	1.0 mg/kg						
C12-C14	1.0	1.0 mg/kg						
C14-C16	1.0	1.0 mg/kg						
C16-C18	1.0	1.0 mg/kg						
C18-C20	1.0	1.0 mg/kg						
C20-C22	1.0	1.0 mg/kg						
C22-C24	1.0	1.0 mg/kg						
C24-C26	1.0	1.0 mg/kg						
C26-C28	1.0	1.0 mg/kg						
C28-C32	1.0	1.0 mg/kg						
C32-C34	1.0	1.0 mg/kg						
C34-C36	1.0	1.0 mg/kg						
C36-C40	1.0	1.0 mg/kg						
C40-C44	1.0	1.0 mg/kg						
TPH (C6-C44)	10	10 mg/kg						
surr: o-Terphenyl			50 - 150					
Diesel Range Organics as Diesel	10	10 mg/kg			70 - 130	40	75 - 125	40

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		Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
Gasoline Range Organics 8015M Preservation: Cool 4°C	M in Soil (EPA	A 8015M)						
Container:10_Metal Sleeve	Cool to 4° C	Amo	ount Required	:250 grams	I	Hold Time:	14 days	
surr: a,a,a-Trifluorotoluene 1,4-Difluorobenzene			80 - 120					
Gasoline Range Organics (GRO)	0.025	0.50 mg/kg			70 - 130	40	75 - 125	40

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		Reporting	Surrogate	Duplicate	Matrix Spike		Blank Spike / LCS	
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
TPH as Stoddard Solvent	(5035) in Soil (EPA	A 8260M/503	5)					
Preservation:Store cool a	at 4°C							
Container:01_40mL P	re-Tared Vial +	Amo	unt Required:	250 grams	Hold Time: 14 days			
10mL MeO	OH; Cool to 4° C							
Stoddard Solvent	0.50	0.50 mg/kg			75 - 125	30	75 - 125	30
Stoddard Sorvein	0.00	0 0						

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix %R	x Spike RPD	Blank Spik %R	e / LCS RPD
8260B/5035 in Soil (EPA 8260I	3/5035)							
Preservation: Store cool at 4°C	C							
Container:07_5g Encore S	ampler Cool	Amo	unt Required	:3-5g encore	H	Iold Time:	14 days	
to 4° C				units				
Acetone	14	50 ug/kg						
Benzene	0.40	2.0 ug/kg			70 - 130	40	75 - 125	40
Bromobenzene	0.70	5.0 ug/kg						
Bromochloromethane	2.0	5.0 ug/kg						
Bromodichloromethane	0.80	5.0 ug/kg					75 - 125	40
surr: 4-Bromofluorobenzene			80 - 120					
Bromoform	2.0	5.0 ug/kg			70 - 130	40	75 - 125	40
Bromomethane	2.0	5.0 ug/kg						40
2-Butanone (MEK)	6.0	50 ug/kg						
sec-Butylbenzene	0.80	5.0 ug/kg						
tert-Butylbenzene	0.70	5.0 ug/kg						
n-Butylbenzene	0.60	5.0 ug/kg						
Carbon Disulfide	4.0	5.0 ug/kg						
Carbon Tetrachloride	0.40	5.0 ug/kg					75 - 125	40
Chlorobenzene	0.40	5.0 ug/kg			70 - 130	40	75 - 125	40
Chlorobenzene-d5								
Chloroethane	0.80	5.0 ug/kg					75 - 125	40
Chloroform	0.80	5.0 ug/kg			70 - 130	40	75 - 125	40
Chloromethane	0.50	5.0 ug/kg					75 - 125	40
2-Chlorotoluene	0.60	5.0 ug/kg						
4-Chlorotoluene	0.50	5.0 ug/kg						
1,2-Dibromo-3-chloropropane	2.0	10 ug/kg						
Dibromochloromethane	2.0	5.0 ug/kg					75 - 125	40
1,2-Dibromoethane (EDB)	2.0	5.0 ug/kg						
surr: Dibromofluoromethane		2 2	80 - 120					
Dibromomethane	2.0	5.0 ug/kg						
1,3-Dichlorobenzene	0.30	5.0 ug/kg						
1,2-Dichlorobenzene	0.60	5.0 ug/kg						
1,4-Dichlorobenzene	0.60	5.0 ug/kg					75 - 125	40
1,4-Dichlorobenzene-d4								
Dichlorodifluoromethane (R12)	0.70	5.0 ug/kg						
1,1-Dichloroethane	0.30	5.0 ug/kg			70 - 130	40	75 - 125	40

		Reporting	Surrogate	Duplicate	Matrix	Spike	Blank Spike / LCS	
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
1,2-Dichloroethane (EDC)	2.0	5.0 ug/kg					75 - 125	40
trans-1,2-Dichloroethylene	2.0	5.0 ug/kg					75 - 125	40
cis-1,2-Dichloroethylene	2.0	5.0 ug/kg			70 - 130	40	75 - 125	40
1,1-Dichloroethylene	3.0	5.0 ug/kg			70 - 130	40	75 - 125	40
Dichlorofluoromethane								
1,3-Dichloropropane	2.0	5.0 ug/kg						
1,2-Dichloropropane	1.0	5.0 ug/kg			70 - 130	40	75 - 125	40
2,2-Dichloropropane	2.0	5.0 ug/kg						
trans-1,3-Dichloropropylene	2.0	5.0 ug/kg						
1,1-Dichloropropylene	0.60	5.0 ug/kg						
cis-1,3-Dichloropropylene	1.0	5.0 ug/kg					75 - 125	40
Ethylbenzene	0.70	2.0 ug/kg			70 - 130	40	75 - 125	40
Hexachlorobutadiene	0.80	10 ug/kg						
2-Hexanone (MBK)	13	50 ug/kg						
Iodomethane	5.0	5.0 ug/kg						
Isopropanol	1000	1000 ug/kg						
Isopropylbenzene	0.50	5.0 ug/kg						
4-Isopropyltoluene	0.50	5.0 ug/kg						
Methyl-tert-Butyl Ether (MTBE)	2.0	5.0 ug/kg			70 - 130	40	75 - 125	40
Methylene Chloride	5.0	50 ug/kg					75 - 125	40
4-Methyl-2-pentanone (MIBK)	6.0	50 ug/kg						
Naphthalene	3.0	10 ug/kg						
Pentafluorobenzene								
n-Propylbenzene	0.70	5.0 ug/kg			70 - 130	40	75 - 125	40
Styrene	0.50	5.0 ug/kg						
1,1,2,2-Tetrachloroethane	2.0	5.0 ug/kg					75 - 125	40
1,1,1,2-Tetrachloroethane	0.70	5.0 ug/kg						
Tetrachloroethylene (PCE)	1.0	5.0 ug/kg			70 - 130	40	75 - 125	40
Tetrahydrofuran (THF)								
Toluene	0.60	2.0 ug/kg			70 - 130	40	75 - 125	40
surr: Toluene-d8			80 - 120					
1,2,3-Trichlorobenzene	2.0	5.0 ug/kg						
1,2,4-Trichlorobenzene	0.90	5.0 ug/kg						
1,1,2-Trichloroethane	2.0	5.0 ug/kg					75 - 125	40
1,1,1-Trichloroethane	0.60	5.0 ug/kg			70 - 130	40	75 - 125	40
Trichloroethylene (TCE)	0.90	5.0 ug/kg			70 - 130	40	75 - 125	40

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		Reporting	ing Surrogate Duplicate		Matrix Spike		Blank Spike / LCS	
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
Trichlorofluoromethane (R11)	3.0	5.0 ug/kg						
1,2,3-Trichloropropane	3.0	5.0 ug/kg						
1,1,2-Trichloro-1,2,2-trifluoroethane	3.0	5.0 ug/kg						
1,2,4-Trimethylbenzene	0.40	5.0 ug/kg						
1,3,5-Trimethylbenzene	0.50	5.0 ug/kg			70 - 130	40		
Vinyl acetate	3.0	5.0 ug/kg						
Vinyl chloride	2.0	5.0 ug/kg			70 - 130	40	75 - 125	40
o-Xylene	0.50	2.0 ug/kg					75 - 125	40
m,p-Xylenes	1.0	2.0 ug/kg						

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix %R	x Spike RPD	Blank Spik %R	e / LCS RPD
8260B in Soil (EPA 8260B)								
Preservation: Store cool at 4°C								
Container:10_Metal Sleeve	Cool to 4° C	Amo	unt Required:	250 grams	H	Iold Time:	:14 days	
Acetone	14	50 ug/kg						
Benzene	0.40	2.0 ug/kg			70 - 130	40	75 - 125	40
Bromobenzene	0.70	5.0 ug/kg						
Bromochloromethane	2.0	5.0 ug/kg						
Bromodichloromethane	0.80	5.0 ug/kg					75 - 125	40
surr: 4-Bromofluorobenzene			80 - 120					
Bromoform	2.0	5.0 ug/kg			70 - 130	40	75 - 125	40
Bromomethane	2.0	5.0 ug/kg						40
2-Butanone (MEK)	6.0	50 ug/kg						
sec-Butylbenzene	0.80	5.0 ug/kg						
n-Butylbenzene	0.60	5.0 ug/kg						
tert-Butylbenzene	0.70	5.0 ug/kg						
Carbon Disulfide	4.0	5.0 ug/kg						
Carbon Tetrachloride	0.40	5.0 ug/kg					75 - 125	40
Chlorobenzene	0.40	5.0 ug/kg			70 - 130	40	75 - 125	40
Chlorobenzene-d5								
Chloroethane	0.80	5.0 ug/kg					75 - 125	40
Chloroform	0.80	5.0 ug/kg			70 - 130	40	75 - 125	40
Chloromethane	0.50	5.0 ug/kg					75 - 125	40
4-Chlorotoluene	0.50	5.0 ug/kg						
2-Chlorotoluene	0.60	5.0 ug/kg						
1,2-Dibromo-3-chloropropane	2.0	10 ug/kg						
Dibromochloromethane	2.0	5.0 ug/kg					75 - 125	40
1,2-Dibromoethane (EDB)	2.0	5.0 ug/kg						
surr: Dibromofluoromethane			80 - 120					
Dibromomethane	2.0	5.0 ug/kg						
1,3-Dichlorobenzene	0.30	5.0 ug/kg						
1,2-Dichlorobenzene	0.60	5.0 ug/kg						
1,4-Dichlorobenzene	0.60	5.0 ug/kg					75 - 125	40
1,4-Dichlorobenzene-d4		- 0						
Dichlorodifluoromethane (R12)	0.70	5.0 ug/kg						
1,1-Dichloroethane	0.30	5.0 ug/kg			70 - 130	40	75 - 125	40

Analyte MDL Limit %R RPD %R RPD %R RPD %R RPD RP			Reporting	Surrogate	Duplicate	Matrix	Spike	Blank Spike / LCS	
1.1-Dichloroethylene	Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
trans-1,2-Dichloroethylene 2.0 5.0 ug/kg 75 - 125 40 cis-1,2-Dichloroethylene 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Dichlorofluoromethane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,2-Dichloropropane 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,3-Dichloropropalene 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 trans-1,3-Dichloropropylene 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 trans-1,3-Dichloropropylene 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 trans-1,3-Dichloropropylene 0.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Hethylenzene 0.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Hexachlorobutadiene 5.0 ug/kg 70 - 130 40 75 - 125 40 Hethyleer-Butyl Ether (MTBE) 2.0 5.0 ug/kg 70	1,2-Dichloroethane (EDC)	2.0	5.0 ug/kg					75 - 125	40
cis1,2-Dichloroethylene 2.0 5.0 ug/kg 70 -130 40 75 -125 40 Dichlorofloromoethane 2.2 S.0 ug/kg 70 -130 40 75 -125 40 1,2-Dichloropropane 1.0 5.0 ug/kg 70 -130 40 75 -125 40 1,3-Dichloropropane 2.0 5.0 ug/kg 70 -130 40 75 -125 40 1,1-Dichloropropylene 1.0 5.0 ug/kg 70 -130 40 75 -125 40 cis-1,3-Dichloropropylene 2.0 5.0 ug/kg 70 -130 40 75 -125 40 Ethylbenzene 0.70 2.0 ug/kg 70 -130 40 75 -125 40 Hexachlorobutadiene 5.0 10 ug/kg 70 -130 40 75 -125 40 2-Hexanore (MBK) 13 50 ug/kg 70 -130 40 75 -125 40 4-Isopropylbeluzene 0.50 5.0 ug/kg 70 -130 40 75 -125 40 Methyl-tert-Butyl Ether (MTBE) 2.0 5.0 ug/kg </td <td>1,1-Dichloroethylene</td> <td>3.0</td> <td>5.0 ug/kg</td> <td></td> <td></td> <td>70 - 130</td> <td>40</td> <td>75 - 125</td> <td>40</td>	1,1-Dichloroethylene	3.0	5.0 ug/kg			70 - 130	40	75 - 125	40
Dichlorofluoromethane	trans-1,2-Dichloroethylene	2.0	5.0 ug/kg					75 - 125	40
2,2-Dichloropropane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,3-Dichloropropane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,3-Dichloropropylene 0.60 5.0 ug/kg 75 - 125 40 40 40 75 - 125 40 40 40 - 75 - 125 40 40 40 - 75 - 125 40 40 40 - 75 - 125 40 40 40 - 75 - 125 40	cis-1,2-Dichloroethylene	2.0	5.0 ug/kg			70 - 130	40	75 - 125	40
1,2-Dichloropropane 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,3-Dichloropropane 2.0 5.0 ug/kg <td< td=""><td>Dichlorofluoromethane</td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></td<>	Dichlorofluoromethane								
1,3-Dichloropropane 2.0 5.0 ug/kg 1,1-Dichloropropylene 0.60 5.0 ug/kg cis-1,3-Dichloropropylene 1.0 5.0 ug/kg Ethylbenzene 0.70 2.0 ug/kg 70 - 130 40 75 - 125 40 Hexachlorobutadiene 0.80 10 ug/kg 70 - 130 40 75 - 125 40 2-Hexachoro (MBK) 13 50 ug/kg 50 50 ug/kg 50 40 75 - 125 40 40 75 - 125 40 40 75 - 125 40 40 75 - 125 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40	2,2-Dichloropropane	2.0	5.0 ug/kg						
1,1-Dichloropropylene	1,2-Dichloropropane	1.0	5.0 ug/kg			70 - 130	40	75 - 125	40
cis-1,3-Dichloropropylene 1.0 5.0 ug/kg 75 - 125 40 Ethylbenzene 0.70 2.0 ug/kg 70 - 130 40 75 - 125 40 Hexachlorobutadiene 0.80 10 ug/kg 70 - 130 40 75 - 125 40 2-Hexanone (MBK) 13 50 ug/kg 70 - 130 40 75 - 125 40 Idodmethane 5.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Isopropylbenzene 0.50 5.0 ug/kg 70 - 130 40 75 - 125 40 Methyl-tert-Butyl Ether (MTBE) 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Methyl-tert-Butyl Ether (MTBE) 5.0 ug/kg 70 - 130 40 75 - 125 40 Methyl-tert-Butyl Ether (MTBE) 5.0 ug/kg 70 - 130 40 75 - 125 40 Methyl-tert-Butyl Ether (MTBE) 5.0 ug/kg 70 - 130 40 75 - 125 40 Hexterlabor (MIBK) 6.0 5.0 ug/kg 70 - 130 40 75 - 125	1,3-Dichloropropane	2.0	5.0 ug/kg						
trans-1,3-Dichloropropylene 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Ekhylbenzene 0.70 2.0 ug/kg 70 - 130 40 75 - 125 40 Hexachlorobutadiene 0.80 10 ug/kg 70 - 130 40 75 - 125 40 2-Hexanone (MBK) 13 50 ug/kg 50 50 ug/kg 50 50 ug/kg 50 50 ug/kg 41-50 propylbenzene 50 5.0 ug/kg 70 - 130 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 75 - 125 40 40 40 45 - 125 40 40	1,1-Dichloropropylene	0.60	5.0 ug/kg						
Ethylbenzene 0.70 2.0 ug/kg 70 - 130 40 75 - 125 40 Hexachlorobutadiene 0.80 10 ug/kg 2-Hexanone (MBK) 13 50 ug/kg Iodomethane 5.0 5.0 ug/kg Isopropylbenzene 0.50 5.0 ug/kg 4-Isopropylbenzene 0.50 5.0 ug/kg Methyl-tert-Butyl Ether (MTBE) 2.0 5.0 ug/kg Methyl-tert-Butyl Ether (MTBE) 5.0 50 ug/kg Methyl-2-pentanone (MIBK) 6.0 50 ug/kg Maphthalene 0.50 5.0 ug/kg Maphthalene 0.50	cis-1,3-Dichloropropylene	1.0	5.0 ug/kg					75 - 125	40
Hexachlorobutadiene 0.80	trans-1,3-Dichloropropylene	2.0	5.0 ug/kg						
2-Hexanone (MBK)	Ethylbenzene	0.70	2.0 ug/kg			70 - 130	40	75 - 125	40
Iodomethane 5.0 5.0 ug/kg 70 - 130 40 75 - 125 40 75 -	Hexachlorobutadiene	0.80	10 ug/kg						
Ropropylbenzene 0.50 5.0 ug/kg 4-lsopropylbenzene 0.50 5.0 ug/kg 70 - 130 40 75 - 125 40 40 40 40 40 40 40 4	2-Hexanone (MBK)	13	50 ug/kg						
4-Isopropyltoluene 0.50 5.0 ug/kg 70 - 130 40 75 - 125 40 Methyl-tert-Butyl Ether (MTBE) 5.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Methylene Chloride 5.0 50 ug/kg 75 - 125 40 4-Methyl-2-pentanone (MIBK) 6.0 50 ug/kg 75 - 125 40 Naphthalene 3.0 10 ug/kg 70 - 130 40 75 - 125 40 Pentafluorobenzene 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Styrene 0.50 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,2,2-Tetrachloroethane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,1,2-Tetrachloroethylene (PCE) 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Surr: Toluene-d8 80 - 120 80 - 120 75 - 125 40 1,2,4-Trichlorobenzene 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,2,3-Trichloroethane 2.0 5.0 ug/kg 75 - 125 40 </td <td>Iodomethane</td> <td>5.0</td> <td>5.0 ug/kg</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>	Iodomethane	5.0	5.0 ug/kg						
Methyl-tert-Butyl Ether (MTBE) 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Methylene Chloride 5.0 50 ug/kg 75 - 125 40 4-Methyl-2-pentanone (MIBK) 6.0 50 ug/kg 75 - 125 40 Naphthalene 3.0 10 ug/kg 70 - 130 40 75 - 125 40 Pentafluorobenzene 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Styrene 0.50 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,2-Tetrachloroethane 0.50 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,1,2-Tetrachloroethane 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrahydrofuran (THF) 80 - 120 70 - 130 40 75 - 125 40 surr: Toluene-d8 80 - 120 5.0 ug/kg 70 - 130 40 75 - 125 40 1,2,3-Trichloroebnae 2.0 5.0 ug/kg 75 - 125 40 75 - 125 40	Isopropylbenzene	0.50	5.0 ug/kg						
Methylene Chloride 5.0 50 ug/kg 75 - 125 40 4-Methyl-2-pentanone (MIBK) 6.0 50 ug/kg 10 ug/kg	4-Isopropyltoluene	0.50	5.0 ug/kg						
4-Methyl-2-pentanone (MIBK) 6.0 50 ug/kg Naphthalene 3.0 10 ug/kg Pentafluorobenzene n-Propylbenzene 0.70 5.0 ug/kg Styrene 0.50 5.0 ug/kg 1,1,2,2-Tetrachloroethane 0.70 5.0 ug/kg Tetrachloroethylene (PCE) 1.0 5.0 ug/kg Tetrahydrofuran (THF) Toluene 0.60 2.0 ug/kg surr: Toluene-d8 1,2,3-Trichlorobenzene 0.90 5.0 ug/kg 1,2,3-Trichloroethane 0.00 5.0 ug/kg 1,1,2,3-Trichloroethane 0.90 5.0 ug/kg 1,1,2-Trichloroethane 0.90 5.0 ug/kg 1,1,1,2-Trichloroethane 0.90 5.0 ug/kg 1,2,3-Trichlorobenzene 0.90 5.0 ug/kg 1,1,1-Trichloroethane 0.60 5.0 ug/kg 1,1,1-Trichloroethane 0.60 5.0 ug/kg 1,1,1-Trichloroethane 0.60 5.0 ug/kg Trichloroethylene (TCE) 0.90 5.0 ug/kg Trichloroethylene (TCE) 0.90 5.0 ug/kg Trichloroethylene (TCE) 0.90 5.0 ug/kg	Methyl-tert-Butyl Ether (MTBE)	2.0	5.0 ug/kg			70 - 130	40	75 - 125	40
Naphthalene 3.0 10 ug/kg Pentafluorobenzene 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Styrene 0.50 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,2,2-Tetrachloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1,2-Tetrachloroethane 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrachloroethylene (PCE) 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrahydrofuran (THF) 80 - 120 80 - 120 70 - 130 40 75 - 125 40 surr: Toluene -d8 80 - 120 80 - 120 1,2,4-Trichlorobenzene 9.90 5.0 ug/kg 70 - 130 40 75 - 125 40 1,2,3-Trichlorobenzene 2.0 5.0 ug/kg 75 - 125 40 1,1,2-Trichloroethane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	Methylene Chloride	5.0	50 ug/kg					75 - 125	40
Pentafluorobenzene n-Propylbenzene 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Styrene 0.50 5.0 ug/kg 75 - 125 40 1,1,2,2-Tetrachloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1,2-Tetrachloroethane 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrachloroethylene (PCE) 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrachloroethylene (PCE) 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrachloroethane 0.60 2.0 ug/kg 70 - 130 40 75 - 125 40 sur: Toluene-d8 80 - 120 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 75 - 125 40 1,1,2-Trichloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	4-Methyl-2-pentanone (MIBK)	6.0	50 ug/kg						
n-Propylbenzene 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Styrene 0.50 5.0 ug/kg 75 - 125 40 1,1,2,2-Tetrachloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1,2-Tetrachloroethane 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrachloroethylene (PCE) 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrahydrofuran (THF) Toluene 0.60 2.0 ug/kg 70 - 130 40 75 - 125 40 surr: Toluene-d8 80 - 120 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,2-Trichloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	Naphthalene	3.0	10 ug/kg						
Styrene 0.50 5.0 ug/kg 1,1,2,2-Tetrachloroethane 2.0 5.0 ug/kg 1,1,1,2-Tetrachloroethane 0.70 5.0 ug/kg Tetrachloroethylene (PCE) 1.0 5.0 ug/kg Tetrahydrofuran (THF) 70 - 130 40 75 - 125 40 surr: Toluene 0.60 2.0 ug/kg 70 - 130 40 75 - 125 40 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 5.0 ug/kg 5.0 ug/kg 75 - 125 40 1,1,2-Trichloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1-Trichloroethane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	Pentafluorobenzene								
1,1,2,2-Tetrachloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1,2-Tetrachloroethane 0.70 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrachloroethylene (PCE) 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrahydrofuran (THF) Toluene 0.60 2.0 ug/kg 70 - 130 40 75 - 125 40 surr: Toluene-d8 80 - 120 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 5.0 ug/kg 5.0 ug/kg 75 - 125 40 1,1,2-Trichloroethane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	n-Propylbenzene	0.70	5.0 ug/kg			70 - 130	40	75 - 125	40
1,1,1,2-Tetrachloroethane 0.70 5.0 ug/kg Tetrachloroethylene (PCE) 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrahydrofuran (THF) Toluene 0.60 2.0 ug/kg 70 - 130 40 75 - 125 40 surr: Toluene-d8 80 - 120 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 5.0 ug/kg 5.0 ug/kg 5.0 ug/kg 75 - 125 40 1,1,2-Trichloroethane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	Styrene	0.50	5.0 ug/kg						
Tetrachloroethylene (PCE) 1.0 5.0 ug/kg 70 - 130 40 75 - 125 40 Tetrahydrofuran (THF) Toluene 0.60 2.0 ug/kg 70 - 130 40 75 - 125 40 surr: Toluene-d8 80 - 120 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 5.0 ug/kg 5.0 ug/kg 5.0 ug/kg 75 - 125 40 1,1,2-Trichloroethane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	1,1,2,2-Tetrachloroethane	2.0	5.0 ug/kg					75 - 125	40
Tetrahydrofuran (THF) Toluene 0.60 2.0 ug/kg 70 - 130 40 75 - 125 40 surr: Toluene-d8 80 - 120 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 1,2,3-Trichlorobenzene 2.0 5.0 ug/kg 1,1,2-Trichloroethane 2.0 5.0 ug/kg 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	1,1,1,2-Tetrachloroethane	0.70	5.0 ug/kg						
Toluene 0.60 2.0 ug/kg 70 - 130 40 75 - 125 40 surr: Toluene-d8 80 - 120 80 - 120 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 5.0 ug/kg 5.0 ug/kg 5.0 ug/kg 75 - 125 40 1,1,2-Trichloroethane 2.0 5.0 ug/kg 70 - 130 40 75 - 125 40 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	Tetrachloroethylene (PCE)	1.0	5.0 ug/kg			70 - 130	40	75 - 125	40
surr: Toluene-d8 80 - 120 1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 1,2,3-Trichlorobenzene 2.0 5.0 ug/kg 1,1,2-Trichloroethane 2.0 5.0 ug/kg 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	Tetrahydrofuran (THF)								
1,2,4-Trichlorobenzene 0.90 5.0 ug/kg 1,2,3-Trichlorobenzene 2.0 5.0 ug/kg 1,1,2-Trichloroethane 2.0 5.0 ug/kg 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	Toluene	0.60	2.0 ug/kg			70 - 130	40	75 - 125	40
1,2,3-Trichlorobenzene 2.0 5.0 ug/kg 1,1,2-Trichloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	surr: Toluene-d8			80 - 120					
1,1,2-Trichloroethane 2.0 5.0 ug/kg 75 - 125 40 1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	1,2,4-Trichlorobenzene	0.90	5.0 ug/kg						
1,1,1-Trichloroethane 0.60 5.0 ug/kg 70 - 130 40 75 - 125 40 Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	1,2,3-Trichlorobenzene	2.0	5.0 ug/kg						
Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	1,1,2-Trichloroethane	2.0	5.0 ug/kg					75 - 125	40
Trichloroethylene (TCE) 0.90 5.0 ug/kg 70 - 130 40 75 - 125 40	1,1,1-Trichloroethane	0.60	5.0 ug/kg			70 - 130	40	75 - 125	40
Trichlorofluoromethane (R11) 3.0 5.0 ug/kg	Trichloroethylene (TCE)	0.90				70 - 130	40	75 - 125	40
	Trichlorofluoromethane (R11)	3.0	5.0 ug/kg						

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			Surrogate Duplicate	Matrix Spike		Blank Spike / LCS	
MDL	Limit	%R	RPD	%R	RPD	%R	RPD
3.0	5.0 ug/kg						
3.0	5.0 ug/kg						
0.40	5.0 ug/kg						
0.50	5.0 ug/kg			70 - 130	40		
3.0	5.0 ug/kg						
2.0	5.0 ug/kg			70 - 130	40	75 - 125	40
0.50	2.0 ug/kg					75 - 125	40
1.0	2.0 ug/kg						
	3.0 3.0 0.40 0.50 3.0 2.0 0.50	3.0 5.0 ug/kg 3.0 5.0 ug/kg 0.40 5.0 ug/kg 0.50 5.0 ug/kg 2.0 5.0 ug/kg 2.0 5.0 ug/kg 0.50 2.0 ug/kg	3.0 5.0 ug/kg 3.0 5.0 ug/kg 0.40 5.0 ug/kg 0.50 5.0 ug/kg 3.0 5.0 ug/kg 2.0 5.0 ug/kg 0.50 2.0 ug/kg	3.0 5.0 ug/kg 3.0 5.0 ug/kg 0.40 5.0 ug/kg 0.50 5.0 ug/kg 3.0 5.0 ug/kg 2.0 5.0 ug/kg 0.50 2.0 ug/kg	3.0 5.0 ug/kg 3.0 5.0 ug/kg 0.40 5.0 ug/kg 0.50 5.0 ug/kg 3.0 5.0 ug/kg 70 - 130 3.0 5.0 ug/kg 2.0 5.0 ug/kg 70 - 130 0.50 2.0 ug/kg	3.0 5.0 ug/kg 3.0 5.0 ug/kg 0.40 5.0 ug/kg 0.50 5.0 ug/kg 70 - 130 40 3.0 5.0 ug/kg 2.0 5.0 ug/kg 70 - 130 40 0.50 2.0 ug/kg	3.0 5.0 ug/kg 3.0 5.0 ug/kg 0.40 5.0 ug/kg 0.50 5.0 ug/kg 2.0 5.0 ug/kg 70 - 130 40 75 - 125 0.50 2.0 ug/kg 75 - 125

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Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix %R	s Spike RPD	Blank Spik %R	e / LCS RPD
8082 PCBs in Soil (EPA 8082 Preservation:Cool 4°C	,							
Container:10_Metal Slee	ve Cool to 4° C	Amo	unt Required:	250 grams	H	Iold Time:	14 days	
Aroclor-1016	2.0	20 ug/kg			50 - 150	40	60 - 140	40
Aroclor-1221	2.0	20 ug/kg						
Aroclor-1232	2.0	20 ug/kg						
Aroclor-1242	2.0	20 ug/kg						
Aroclor-1248	2.0	20 ug/kg						
Aroclor-1254	2.0	20 ug/kg						
Aroclor-1260	2.0	20 ug/kg			50 - 150	40	60 - 140	40
surr: Tetrachloro-meta-xylene			50 - 150					
surr: Decachlorobiphenyl			50 - 150					

		Reporting	Surrogate	Duplicate	Matrix	Spike	Blank Spike / LCS	
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
CAM Metals Less Hg (Preservation: Cool 4°	•	6010B/7000))					
Container:10_Met	al Sleeve Cool to 4° C	Amo	unt Required:	250 gm	H	lold Time:	180 days	
Antimony	10	10 mg/kg			75 - 125	40	80 - 120	20
Arsenic	0.50	0.50 mg/kg			75 - 125	40	80 - 120	20
Barium	10	10 mg/kg			75 - 125	40	80 - 120	20
Beryllium	1.0	1.0 mg/kg			75 - 125	40	80 - 120	20
Cadmium	1.0	1.0 mg/kg			75 - 125	40	80 - 120	20
Chromium	3.0	3.0 mg/kg			75 - 125	40	80 - 120	20
Cobalt	3.0	3.0 mg/kg			75 - 125	40	80 - 120	20
Copper	3.0	3.0 mg/kg			75 - 125	40	80 - 120	20
Lead	3.0	3.0 mg/kg			75 - 125	40	80 - 120	20
Molybdenum	5.0	5.0 mg/kg			75 - 125	40	80 - 120	20
Nickel	3.0	3.0 mg/kg			75 - 125	40	80 - 120	20
Selenium	0.50	0.50 mg/kg			75 - 125	40	80 - 120	20
Silver	1.0	1.0 mg/kg			75 - 125	40	80 - 120	20
Thallium	5.0	5.0 mg/kg			75 - 125	40	80 - 120	20
Tin	10	10 mg/kg				40		20
Vanadium	10	10 mg/kg			75 - 125	40	80 - 120	20
Zinc	3.0	3.0 mg/kg			75 - 125	40	80 - 120	20

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		Reporting	Surrogate	Duplicate	Matrix	Spike	Blank Spik	e / LCS
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
Mercury Total EPA 74 Preservation:Cool 4°	,	'A 7471A)						
Container:10_Met	tal Sleeve Cool to 4° C	Amo	unt Required:	250 gm	Hold Time: 28 days			
Mercury	0.00090	0.020 mg/kg		25	75 - 125	25	85 - 115	25

Analyte	MDL	Reporting Limit	Surrogate %R	Duplicate RPD	Matrix %R	x Spike RPD	Blank Spik %R	e / LCS RPD
8270C in Soil (EPA 8270C) Preservation:Cool 4°C Container:10_Metal Sleeve Cool to 4° C		Amo	Amount Required:250 gm		Hold Time		:14 days	
3,3'-Dichlorobenzidine	0.30	0.40 mg/kg						
Acenaphthene	0.050	0.10 mg/kg			47 - 145	40	50 - 121	40
Acenaphthene-d10								
Acenaphthylene	0.050	0.10 mg/kg						
Aniline	0.050	0.20 mg/kg						
Anthracene	0.040	0.10 mg/kg			27 - 133	40	41 - 121	40
Azobenzene	0.040	0.10 mg/kg						
Benzidine	0.20	0.40 mg/kg						
Benzo(a)anthracene	0.060	0.10 mg/kg						
Benzo(a)pyrene	0.040	0.10 mg/kg			17 - 163	40	17 - 163	40
Benzo(b)fluoranthene	0.060	0.10 mg/kg			24 - 159	40	25 - 137	40
Benzo(g,h,i)perylene	0.050	0.10 mg/kg						
Benzoic acid	0.30	1.0 mg/kg						
Benzo(k)fluoranthene	0.030	0.10 mg/kg						
Benzyl alcohol	0.070	0.10 mg/kg						
4-Bromophenyl phenyl ether	0.060	0.10 mg/kg						
Butyl benzyl phthalate	0.070	0.50 mg/kg			2 - 152	40	19 - 139	40
4-Chloro-3-methylphenol	0.050	0.20 mg/kg			22 - 147	40	22 - 147	40
4-Chloroaniline	0.20	0.40 mg/kg						
Bis(2-chloroethoxy)methane	0.040	0.10 mg/kg						
Bis(2-chloroethyl)ether	0.010	0.10 mg/kg			12 - 158	40	26 - 122	40
Bis(2-chloroisopropyl)ether	0.010	0.10 mg/kg						
2-Chloronaphthalene	0.050	0.10 mg/kg			60 - 118	40	60 - 118	40
2-Chlorophenol	0.020	0.10 mg/kg						
4-Chlorophenyl phenyl ether	0.070	0.10 mg/kg			25 - 158	40	41 - 128	40
Chrysene	0.050	0.10 mg/kg						
Chrysene-d12								
Dibenzo(a,h)anthracene	0.10	0.10 mg/kg						
Dibenzofuran	0.050	0.10 mg/kg						
Di-n-butyl phthalate	0.080	2.0 mg/kg						
1,2-Dichlorobenzene	0.010	0.10 mg/kg						
1,3-Dichlorobenzene	0.010	0.10 mg/kg						
		- 0						

			Surrogate %R	Duplicate RPD	Matrix Spike		Blank Spike / LCS	
Analyte	MDL				%R	RPD	%R	RPD
1,4-Dichlorobenzene	0.010	0.10 mg/kg			20 - 124	40	26 - 105	40
1,4-Dichlorobenzene-d4								
2,4-Dichlorophenol	0.10	0.10 mg/kg			39 - 135	40	39 - 135	40
Diethyl phthalate	0.080	0.80 mg/kg						
2,4-Dimethylphenol	0.10	0.10 mg/kg						
Dimethyl phthalate	0.080	0.20 mg/kg						
4,6-Dinitro-2-methylphenol	0.090	0.20 mg/kg						
2,4-Dinitrophenol	0.080	0.40 mg/kg						
2,6-Dinitrotoluene	0.060	0.10 mg/kg						
2,4-Dinitrotoluene	0.060	0.10 mg/kg						
Di-n-octyl phthalate	0.090	0.10 mg/kg			4 - 146	40	4 - 146	40
1,2-Diphenylhydrazine	0.050	0.10 mg/kg						
Bis(2-ethylhexyl)phthalate	0.10	0.20 mg/kg						
Fluoranthene	0.050	0.10 mg/kg			26 - 137	40	35 - 125	40
Fluorene	0.050	0.10 mg/kg			59 - 121	40	50 - 120	40
surr: 2-Fluorobiphenyl			43 - 116					
surr: 2-Fluorophenol			21 - 100					
Hexachlorobenzene	0.020	0.10 mg/kg			2 - 152	40	2 - 152	40
Hexachlorobutadiene	0.020	0.10 mg/kg			24 - 116	40	24 - 116	40
Hexachlorocyclopentadiene	0.040	0.10 mg/kg						
Hexachloroethane	0.060	0.10 mg/kg			40 - 113	40	40 - 113	40
Indeno (1,2,3-cd) pyrene	0.20	0.40 mg/kg						
Isophorone	0.030	0.10 mg/kg			21 - 196	40	21 - 196	40
2-Methylnaphthalene	0.030	0.10 mg/kg						
2-Methylphenol	0.030	0.20 mg/kg						
3-Methylphenol	0.050	0.20 mg/kg						
4-Methylphenol	0.030	0.20 mg/kg						
Naphthalene	0.040	0.10 mg/kg			21 - 133	40	25 - 121	40
Naphthalene-d8								
4-Nitroaniline	0.40	0.50 mg/kg						
3-Nitroaniline	0.060	0.40 mg/kg						
2-Nitroaniline	0.080	0.10 mg/kg						
Nitrobenzene	0.030	0.10 mg/kg			35 - 180	40	38 - 133	40
surr: Nitrobenzene-d5		- 0	35 - 134					
2-Nitrophenol	0.040	0.20 mg/kg			2 - 163	40	2 - 163	40
4-Nitrophenol	0.20	0.20 mg/kg						

		Reporting	Surrogate	Duplicate	Matrix	x Spike	Blank Spik	e / LCS
Analyte	MDL	Limit	%R	RPD	%R	RPD	%R	RPD
N-Nitrosodimethylamine	0.040	0.10 mg/kg						
N-Nitrosodiphenylamine	0.050	0.10 mg/kg						
N-Nitrosodi-n-propylamine	0.040	0.10 mg/kg			2 - 230	40	2 - 230	40
Pentachlorophenol	0.10	0.10 mg/kg			14 - 176	40	14 - 176	40
Perylene-d12								
Phenanthrene	0.030	0.10 mg/kg						
Phenanthrene-d10								
Phenol	0.010	0.10 mg/kg			5 - 112	40	5 - 112	40
surr: Phenol-d6			10 - 94					
Pyrene	0.040	0.10 mg/kg			52 - 115	40	52 - 115	40
surr: Terphenyl-dl4			33 - 141					
surr: 2,4,6-Tribromophenol			10 - 123					
1,2,4-Trichlorobenzene	0.020	0.10 mg/kg			44 - 142	40	44 - 142	40
2,4,5-Trichlorophenol	0.030	0.20 mg/kg						
2,4,6-Trichlorophenol	0.040	0.20 mg/kg			37 - 144	40	37 - 144	40

QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL ANALYTICAL SERVICES



Version 5.0 January 2007

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Based On

National Environmental Laboratory Accreditation Program (NELAP)
Chapter 5 (Quality Systems)
NELAC Standard Effective July 01, 2004

JALITY ASSURANCE MANAGER

Virginia Huang TECHNICAL DIRECTOR

Steven L. Lane
LABORATORY DIRECTOR

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PREFACE TO THE QUALITY SYSTEMS MANUAL

Purpose

The purpose of this document is to provide implementation guidance on the establishment and management of quality systems for Calscience Environmental Laboratories, Inc and is based on the National Environmental Laboratory Accreditation Conference's (NELAC) Quality System requirements.

Background

To be accredited under the National Environmental Laboratory Accreditation Program (NELAP), laboratories shall have a comprehensive quality system in place, the requirements for which are outlined in NELAP Chapter 5 (Quality Systems).

Project Specific Requirements

Project-specific requirements or regulations may supersede requirements contained in this manual. The laboratory bears the responsibility for meeting all **State requirements**. Nothing in this document relieves the laboratory from complying with contract requirements, or with **Federal**, **State**, **and/or local regulations**.

Results and Benefits

- Standardization of Processes Because this manual provides the laboratory with a comprehensive set of requirements that meet the needs of many clients, as well as the NELAP, the laboratory may use it to create a standardized quality system. Ultimately, this standardization saves laboratory resources by establishing one set of consistent requirements for all environmental work. Primarily, the laboratory bears the responsibility for meeting all State requirements as outlined in their respective certification programs.
- **Deterrence of Improper, Unethical, or Illegal Actions** Improper, unethical, or illegal activities committed by only a few laboratories have implications throughout the industry, with negative impacts on all laboratories. This manual establishes a minimum threshold program for all laboratories to use to deter and detect improper, unethical, or illegal actions.
- Foundations for the Future A standardized approach to quality systems, shared by laboratories and the NELAP, paves the way for the standardization of other processes. For example, this manual might serve as a platform for a standardized strategy for Performance Based Measurement System (PBMS) implementation.

Document Format

This Calscience Environmental Laboratories, Inc. (Calscience) *Quality Systems Manual* (QSM) is designed to implement NELAP Chapter 5 (Quality Systems) and the NELAP Chapter 5 document serves as the primary text for this implementation manual. This Calscience QMS is also a complement to NELAP chapter 5. The section numbering has been changed from that of NELAP Chapter 5 as the manual is meant to be a stand-alone document. The number 5 has been eliminated from all section and subsection headings. However, second-level numbering has been retained to ensure maintenance of a parallel organization to the NELAC Quality Systems requirements. For instance, Section 5.4.2 in NELAP Chapter 5 (referencing Chapter 5 of the NELAC standards) is equivalent to Section 4.2 in this manual. In addition, there is one set of NELAC appendices.

ACROYNM LIST

°C: Degrees Celsius

ANSI/ASQC: American National Standards Institute/American Society for Quality Control

ASTM: American Society for Testing and Materials

CAS: Chemical Abstract Service
CCV: Continuing calibration verification
CFR: Code of Federal Regulations
CLP: Contract Laboratory Program

COC: Chain of custody
CV: Coefficient of variation
DO: Dissolved oxygen

DOC: Demonstration of capability **DQOs:** Data quality objectives

EPA: Environmental Protection Agency

g/L: Grams per liter

GC/MS: Gas chromatography/mass spectrometry **ICP-MS:** Inductively coupled plasma-mass spectrometer

ICV: Initial calibration verification

ID: Identifier

ISO/IEC: International Standards Organization/International Electrotechnical Commission

LCS: Laboratory control sample

LCSD: Laboratory control sample duplicate **LQMP:** Laboratory Quality Management Plan

MDL: Method detection limit **mg/kg:** Milligrams per kilogram

MS: Matrix spike

MSD: Matrix spike duplicate

NELAC: National Environmental Laboratory Accreditation Conference **NELAP:** National Environmental Laboratory Accreditation Program

NIST: National Institute of Standards and Technology **OSHA:** Occupational Safety and Health Administration **PBMS:** Performance Based Measurement System

PC: Personal computer

PCBs: Polychlorinated biphenyls

PT: Proficiency testing **QA:** Quality assurance

QAD: Quality Assurance Division (EPA)

QAMS: Quality Assurance Management Section

QAPP: Quality Assurance Project Plan

QSM: Quality Systems Manual

QC: Quality control RL: Reporting limit

RPD: Relative percent difference **RSD:** Relative standard deviation

SD: Serial dilutions

SOP: Standard operating procedure

TSS: Total suspended solids

UV: Ultraviolet

VOC: Volatile organic compound

QUALITY SYSTEMS

Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures that are delineated in a Quality Systems Manual (QSM) and followed to ensure and document the quality of the analytical data. Calscience, accredited under the National Environmental Accreditation Program (NELAP), assures implementation of all QA policies and the applicable QC procedures specified in this Manual. The QA policies, which establish essential QC procedures, are applicable to all areas of Calscience, regardless of size and complexity.

The intent of this document is to provide sufficient detail about quality management requirements so that all accrediting authorities evaluate laboratories consistently and uniformly.

The National Environmental Laboratory Accreditation conference (NELAC) is committed to the use of Performance Based Measurement Systems (PBMS) in environmental testing and provides the foundation for PBMS implementation in these standards. While this standard may not currently satisfy all the anticipated needs of PBMS, NELAC will address future needs within the context of State statutory and regulatory requirements and the finalized EPA implementation plans for PBMS.

Chapter 5 is organized according to the structure of ISO/IEC 17025, 1999. Where deemed necessary, specific areas within this Chapter may contain more information than specified by ISO/IEC 17025.

All items identified in this QSM shall be available for on-site inspection or data audit.

1.0 SCOPE

- a) This QSM sets the general requirements that Calscience must successfully demonstrate to be recognized as competent to perform specific environmental tests.
- b) This QSM includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority that grants approval.
 - If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory demonstrates that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed.
- c) Calscience uses this QSM in the development and implementation of its quality systems. Accreditation authorities use this NELAC based standard to assess the competence of environmental laboratories.

2.0 REFERENCES

See Appendix A.

3.0 DEFINITIONS

The relevant definitions from ISO/IEC Guide 2, ANSI/ASQC E-4, 1994, the EPA "Glossary of Quality Assurance Terms and Acronyms," and the *International vocabulary of basic and general terms in metrology (VIM)* are applicable. The most relevant is quoted in Appendix A, Glossary, of Chapter 1 of NELAC, together with further definitions applicable for the purposes of this Standard.

4.0 ORGANIZATION AND MANAGEMENT

4.1 Legal Definition of Laboratory

Calscience is legally definable as evidenced by its business license, and current California Department of Health Services Environmental Laboratory Accreditation Program (CADHS ELAP) certificate. It is organized and operates in such a way that its facilities meet the requirements of the Standard. See the graphical presentations of the Organization and QA responsibility in Figures 1 and 2, respectively.

4.2 Organization

Calscience:

- a) Has a managerial staff with the authority and resources necessary to discharge their duties;
- b) Has processes to ensure that its personnel are free from any commercial, financial and other undue pressure that adversely affect the quality of their work;
- c) Is organized in such a way that confidence in its independence of judgment and integrity is maintained at all times;
- d) Specifies and documents the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests;

Such documentation includes:

- 1) A clear description of the lines of responsibility in the laboratory, and is proportioned such that adequate supervision is ensured, and
- 2) Job descriptions for all positions.
- e) Provides supervision by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test, and the assessment of the results.

The ratio of supervisory to non-supervisory personnel ensures adequate supervision and adherence to laboratory procedures and accepted techniques.

f) Has a technical director who has overall responsibility for the technical operation of Calscience.

The technical director certifies that personnel who perform the tests for which the laboratory is accredited have the appropriate educational and/or technical background. Such certification is documented.

The technical director meets the requirements specified in the Accreditation Process. (See NELAC Section 4.1.1.1.)

g) Has a quality assurance manager who has responsibility for the quality system and its implementation.

The quality assurance officer has direct access to the technical director and to the highest level of management at which decisions are made regarding laboratory policy or resources.

The quality assurance manager (and/or his/her designees):

- Serves as the focal point for QA/QC activities, and is responsible for the oversight and/or review of quality control data;
- Has functions independent from laboratory operations for which she/he has quality assurance oversight;
- Is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence:
- 4) Has documented training and/or experience in QA/QC procedures and is knowledgeable in the quality system, as defined under NELAC;
- 5) Has a general knowledge of the analytical test methods for which data review is performed;
- 6) Arranges for and conduct internal audits as per Calscience QSM section 5.3 annually; and
- 7) Notifies Calscience management of deficiencies in the quality system and monitors corrective action.
- h) Nominates, by way of the "Alternates List," deputies in case of absence of the technical director and/or the quality assurance officer;
- i) Calscience makes every effort to ensure the protection of its clients' information as confidential and proprietary.
 - ii) Calscience is sensitive to the fact that much of the analytical work performed for clientele may be subject to litigatory processes. Calscience, therefore, holds all information in strict confidence with laboratory release only to the client.
 - iii) Information released to entities other than the client is performed only upon written request from the client.
 - iv) Due to the investigative nature of most site assessments, analytical information may become available to regulatory agencies or other evaluating entities during site assessment of the laboratory for the specific purpose of attaining laboratory certifications, accreditations, or evaluation of laboratory qualification for future work. During these occurrences, the laboratory will make every effort to maintain the confidence of client specific information.
- j) For purposes of qualifying for and maintaining accreditation, participates in a proficiency test program as outlined in Chapter 2 of NELAC. Results of Calscience's performance in rounds of proficiency testing are available by request.

5.0 QUALITY SYSTEM – ESTABLISHMENT, AUDITS, ESSENTIAL QUALITY CONTROLS, AND DATA VERIFICATION

5.1 Establishment

Calscience establishes and maintains quality systems based on the required elements contained in this Manual and appropriate to the type, range and volume of environmental testing activities it undertakes.

- a) The elements of this quality system are documented in this quality manual.
- b) The quality documentation is available for use by all laboratory personnel.
- c) The laboratory defines and documents its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

- d) The laboratory management ensures that these policies and objectives are documented in the quality manual and are communicated to, understood and implemented by all laboratory personnel concerned.
 - i. All staff members are issued a copy of the quality manual at the commencement of work at Calscience. Employees read and endorse the following statement when they receive their quality manual: "By signature below, I acknowledge that I have received a copy of Revision [number] of Calscience's Quality Assurance Program Manual dated [effective date of the subject manual]. Furthermore, I agree to read and abide by the policies contained therein."
 - ii. A controlled copy of the quality manual is also available at the designated data reduction areas.
- e) The quality manual is maintained current under the responsibility of the quality assurance officer. This manual is reviewed on an annual basis or more frequently, and revised as necessary. The review process begins in January of each year, and concludes on/before March of the same year. Where no revision is required, the manual is reissued in its entirety and review is scheduled for January of the following year.

5.2 Quality Systems Manual (QSM)

This quality systems manual and related quality documentation state Calscience's policies and operational procedures established in order to meet the requirements of this Standard.

This Manual lists on the title page: a document title; the laboratory's full name and address; the name, address, and telephone number of individuals responsible for the laboratory; the name of the quality assurance manager; the identification of all major organizational units that are covered by this quality manual and the effective date of the version.

This quality manual and related quality documentation also contains:

- A quality policy statement, including objectives and commitments, by top management;
 - i. Calscience Environmental Laboratories, Inc. (Calscience) is committed to providing the highest quality environmental analytical services available. To ensure the production of scientifically sound, legally defensible data of known and proven quality, an extensive Quality Assurance program has been developed and implemented. This document, Calscience's <u>Quality Systems Manual for Environmental Analytical Services</u>, presents an overview of the essential elements of our Quality Assurance program. Calscience has modeled this systems manual after EPA guidelines as outlined in "<u>Guidance for Quality Assurance Project Plans (EPA QA/G-5)</u>", Office of Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. EPA, EPA/240-R-02/009 December 2002. Calscience's QA Program is closely monitored at the Corporate, Divisional, and Group levels, and relies on clearly defined objectives, well-documented procedures, a comprehensive quality assurance/quality control system, and management support for its effectiveness.
 - ii. This QA Program Systems Manual is designed to control and monitor the quality of data generated at Calscience. The essential elements described herein are geared toward generating data that is in compliance with federal regulatory requirements specified under the Clean Water Act, the Safe Drinking Water Act, the Resource Conservation and Recovery Act, the Comprehensive Environmental Response, Compensation, and Liability Act, and applicable amendments, and state and DoD/DoE equivalents. Although the quality control requirements of these various programs are not completely consistent, each of the programs base data quality

judgments on the following three types of information, the operational elements of each being described elsewhere in this manual.

- ⇒ Data which indicates the overall qualifications of the laboratory to perform environmental analyses;
- ⇒ Data which measures the laboratory's daily performance using a specific method; and
- ⇒ Data which measures the effect of a specific matrix on the performance of a method.
- iii. It is important to note that the QA guidelines presented herein will always apply unless adherence to specific Quality Assurance Project Plans (QAPPs) or client and/or regulatory agency specific requirements are directed. In these cases, the elements contained within specified direction or documentation shall supersede that contained herein.
- iv. This manual is a living document subject to periodic modifications to comply with regulatory changes and technological advancements. All previous versions of this document are obsolete. Users are urged to contact Calscience to verify the current revision of this document.
- b) The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;
 - See Figure 1 Organizational Chart, and Figure 2 QA Responsibility Chart.
- c) The relationship between management, technical operations, support services and the quality system;
- d) Procedures to ensure that all records required under the NELAP are retained, as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;
 - i. Ensuring a high quality work product in the environmental laboratory not only requires adherence to the quality issues discussed in the previous sections, but also requires the ability to effectively archive, restore, and protect the records that are generated.
 - ii. Procedures are in place to ensure that all records are retained. In addition, a documentation control system is employed to clearly indicate the time period during which a standard operating procedure, manual, or document was in force. These procedures are outlined in the laboratory standard operating procedure SOP-T002.
 - iii. All laboratory logbooks, instrument response printouts, completed analytical reports, chain-of-custodies, and laboratory support documentation are stored for a minimum of five years. Project specific data are stored in sequentially numbered project files and include copies of the applicable laboratory logbooks, instrument response printouts, completed analytical reports, chain-of-custodies, and any other pertinent supporting documentation.
 - iv. When complete, the project specific data are high speed optically scanned and transformed into digital CD media. Additional copies of these records are created at the time of scanning and are stored off-site for protection of the data. These records are stored for a minimum of five years.

FIGURE 1: ORGANIZATIONAL CHART

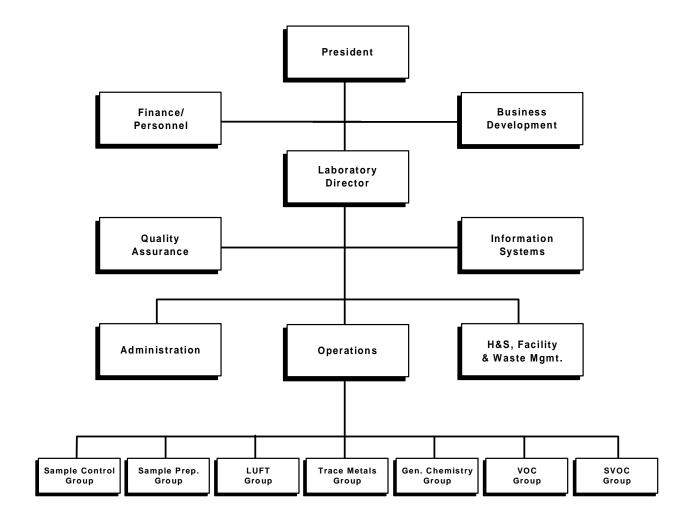
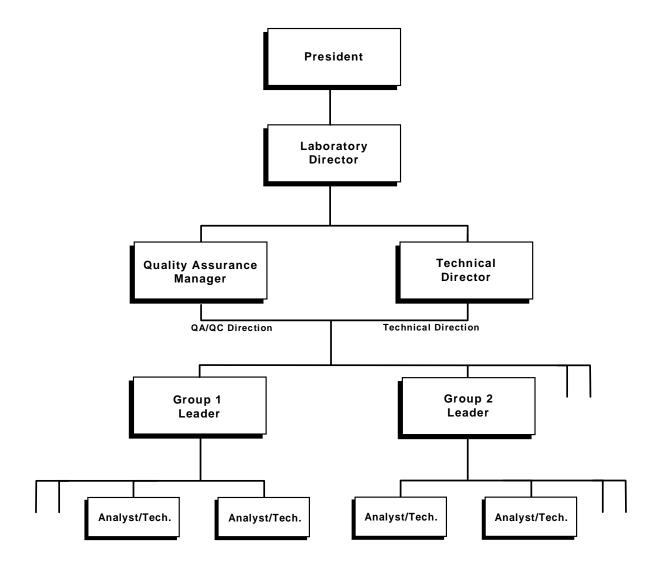


FIGURE 2: QA RESPONSIBILITY CHART



v. Access to all systems is limited by use of log-in and password protection and is maintained by the system administrator.

There are four forms of electronic data that are generated in the laboratory and a synopsis of the archiving of these data follows:

LIMS Database

Backup frequency: Daily
Backup media: Hard Disk

Backup software: MS SQL Server Backup Backup versions kept: Ten previous versions

Onsite copy: Redundancy by using mirrored hard drive

Instrument Data

Backup frequency: Weekly

Backup media: Quantum 4000 DLT Raid Tape and DLT Tape

Backup software: Computer Associates ArcServIT

Backup versions kept: All versions
Offsite copy: One

Manual Data

Backup frequency: Weekly

Backup media: Quantum 4000 DLT Raid Tape and DLT Tape

Backup software: Computer Associates ArcServIT

Backup versions kept: All versions
Offsite copy: One

Hard Copy Data

Backup media: Digital CD
Backup software: Xerox Pagis

Backup versions kept: All versions
Offsite copy: One

- vi. All electronic records are stored for a minimum of five years.
- e) Job descriptions of key staff and reference to the job descriptions of other staff;
 - i. Calscience's Laboratory Director, through its President, is the final authority on all issues dealing with data quality and has the authority to require that procedures be amended or discontinued, or analytical results voided or repeated. He or she also has the authority to suspend or terminate employees on the grounds of non-compliance with QA/QC procedures. In addition, the Laboratory Director:
 - ⇒ Ensures that Calscience remains current with all regulations which affects operations and disseminate all such changes in regulatory requirements to the QA Manager, Technical Director, and Group Leaders;
 - Develops and implements Calscience's QA Program which assures that all data generated will be scientifically sound, legally defensible, and of known precision and accuracy;
 - ⇒ Conducts annual reviews of Calscience's QA Program;
 - ⇒ Routinely monitors the QA Program to ensure compliance;
 - ⇒ Develops and implement new and revised QA procedures to improve data quality;

 - ⇒ Develop and implement project specific QA plans (QAPPs); and
 - ⇒ Monitor in-house training on quality assurance and control.

- Develops and implements laboratory policy in order to review all new work and ensure that it has the appropriate facilities and resources to complete such work.
- ii. The QA Manager has full authority through the Laboratory Director in matters dealing within the laboratory. The QA Manager can make recommendations to the Laboratory Director regarding the suspension or termination of employees on the grounds of non-compliance with QA/QC procedures. An alternate QA Manager is always assigned. In the absence of the primary designate, the alternate will act in the QA Manager's capacity with the full authority of the position as allowed by Calscience governing documents. In addition, the QA Manager performs the following:
 - ⇒ Implements Calscience's QA Program;
 - ➡ Monitors the QA Program within the laboratory to ensure complete compliance with its objectives, QC procedures, holding times, and compliance with client or project specific data quality objectives;
 - Distributes performance evaluation (PE) samples on a routine basis to ensure the production of data that meets the objectives of its QA Program;
 - ⇒ Maintains all SOPs used at Calscience:

 - Performs statistical analyses of QC data and establish controls that accurately reflect the performance of the laboratory;
 - ⇔ Conducts periodic performance and system audits to ensure compliance with the elements of Calscience's QA Program;
 - ⇒ Prescribes and monitor corrective action;
 - ⇒ Serves as in-house client representative on all project inquiries involving data quality issues;
 - ⇔ Coordinates data review process to ensure that thorough reviews are conducted on all project files:
 - ⇒ Develops revisions to existing SOPs;
 - ⇒ Reports the status of in-house QA/QC to the Laboratory Director:
 - ⇒ Distributes new SOPs to all applicable lab areas;
 - ⇒ Maintains records and archives of all QA/QC data including but not limited to method detection limit (MDL) studies, accuracy and precision control charts, and completed log books; and
 - ⇒ Conducts and/or otherwise ensures that an adequate level of QA/QC training is conducted within the laboratory.
- iii. The Technical Director has full authority through the Laboratory Director in matters dealing with technical proceedings within the laboratory. He or she can make recommendations to the Laboratory Director regarding the suspension or termination of employees on the grounds of non-compliance with QA/QC procedures. The Technical Director also
 - ⇒ Implements Calscience's training program to ensure that all personnel are properly trained for the tasks being performed;
 - ⇒ Resolves technical difficulties encountered during normal operations;
 - ⇒ Oversees all method developmental activities within Calscience;
 - ⇒ Ensures compliance with approved methodologies, standard operating procedures (SOPs), this manual, QAPPs, and all other governing documents; and
 - ⇒ Implements a system of continual improvement within Calscience, to include reviews of new technologies that may potentially improve quality.
- iv. The Group Leaders have the authority to accept or reject data based on pre-defined QC criteria. In addition, with the approval of the QA Manager, the Group Leaders may accept data that falls outside of normal QC limits if, in his or her professional judgment, there are technical justifications for the acceptance of such data. The circumstances must be well documented and any need for corrective action identified must be defined and initiated. The authority of the Group Leaders in QC related matters results directly from the QA Manager. The Group Leaders also
 - ⇒ Actively support the implementation of Calscience's QA Program;
 - ⇒ Ensure that their employees are in full compliance with Calscience's QA Program;

- Maintain accurate (by recommending changes to) SOPs and enforce routine compliance with SOPs:
- Conduct technical training of new staff and when modifications are made to existing procedures;
- ⇒ Perform secondary QC reviews on all data generated within their respective groups;
- ⇒ Maintain a work environment which emphasizes the importance of data quality; and
- ⇒ Provide support to all levels of Calscience Management.
- Laboratory staff members have the authority to accept or reject data based on compliance with well-defined QC acceptance criteria. Their supervisor must approve the acceptance of data that falls outside the QC criteria.

 - ⇒ Ensure that all data is generated in compliance with Calscience's QA Program;
 - ⇒ Perform work in strict accordance with the SOPs;
 - ⇒ Ensure that all documentation related to their work is complete, accurate, and legible; and
 - ⇒ Immediately inform their supervisors of data quality problems.

vi. Project Managers

- ⇒ Maintain a working knowledge of Calscience's QA Program;
- ⇒ Verify that all final reports are in compliance with predetermined client- and/or project-specific criteria;
- ⇒ Ensure that all supporting documentation to a specific report is complete, accurate, and legible; and
- Effectively track and implement systems that ensure the best available service to Calscience's customers.
- f) Identification of the laboratory's approved signatories; at a minimum, the title page of the quality manual has the signed and dated concurrence (with appropriate titles) of all responsible parties including the QA manager, technical director, and the laboratory director;
- g) The laboratory's procedures for achieving traceability of measurements;
- h) A list of all test methods under which the laboratory performs its accredited testing may be found in the Index of Standard Operating Procedures, a separate document.
- i) Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;
- i) Reference to the calibration and/or verification test procedures used;
 - Calibration procedures and verification of acceptability for each set of required calibrations are defined in Section 13 (Calibration) and Section 12 (Quality Control) of each standard operating procedure.
- k) Procedures for handling samples received;

The generation of quality analytical data begins with the collection of the sample and, therefore, the integrity of the sample collection process is of importance to Calscience. Samples must be collected in such a way that foreign material is not introduced into the samples and that analytes of interest do not escape from the samples or degrade prior to their analysis. To ensure sample integrity and representativeness, the following items must be considered:

⇒ Samples must be collected in appropriate containers. In general, glass containers are used for organic analytes and polyethylene for inorganic/metal analytes;

- ⇒ Only new sample containers which are certified and documented clean in accordance with U.S. EPA OSWER Directive No. 9240.0-0.05 specifications shall be provided by Calscience for sample collection;
- ⇒ Certain extremely hazardous samples or samples that have the potential to become extremely hazardous will not be accepted. These include (but are not limited to)
 - 1. Radioactive samples that exceed background levels
 - 2. Biohazardous samples (medical wastes, body fluids, etc.)
 - 3. Explosive samples (Flash or gunpowder, ammunition, flares, etc.)
 - 4. Neurological or other toxic agents (Sarin, Anthrax, Ricin, etc.)

Calscience's chain-of-custody document is used to forward samples from the client to the laboratory. As the basic elements of most all chain-of-custody (COC)documents are similar, clientele may choose to use their own chain-of-custody document to forward samples to Calscience.

Any discrepancies in the COC must be documented on the Sample Receipt Form and resolved prior to analysis of samples. Further guidance may be found in SOP T100 "Sample Receipt and Log-In Procedures".

Upon receipt by Calscience, samples proceed through an orderly processing sequence designed to ensure continuous integrity of both the sample and its documentation from sample receipt through its analysis and beyond.

All coolers that are received by the Sample Control Group undergo a preliminary examination in accordance with Part A of the Sample Receipt Form. Specifically, each sample is carefully examined for label identification, proper container (type and volume), chemical preservation when applicable, container condition, and chain-of-custody documentation consistency with sample labels. Discrepancies are noted on the Sample Receipt Form, the chain-of-custody and, if possible, discussed with the client prior to his or her departure. If this is not possible, the discrepancies are communicated to the client for resolution prior to the completion of the log-in process. The temperature of the cooler is measured and, with other observations, is recorded in Part B of the Sample Receipt Form. Additional comments are recorded in Part C of the Sample Receipt Form.

During the log-in process each sample is assigned a unique laboratory identification number through a computerized Laboratory Information Management System (LIMS), which stores all essential project information. Calscience maintains multiple security levels of access into LIMS to prevent unauthorized tampering/release of sample and project information.

Once all analyses for a sample have been completed and the sample container is returned to Sample Control, it shall remain in refrigerated storage for a period not less than 30 days following sample receipt unless the client requests return/forwarding of the sample. Following the 30-day refrigerated storage period, the samples are placed into ambient storage for another period not less than 30 days after which the samples are bulked into drums for later disposal.

Extended storage may be requested at prevailing per sample rates.

- I) Reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;
 - A list of major equipment is kept up-to-date on the List of Major Assets. This, as well as a list of reference measurement standards and their certificates of calibration, is maintained by the QA Manager or in the respective departments.
- m) Reference to procedures for calibration, verification and maintenance of equipment; Laboratory SOPs (T042, T050 and T051) are available to staff for calibration, verification and maintenance of equipment.

n) Reference to verification practices which may include interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

Instrument calibration is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity such that required reporting limits can be met. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method. The manufacturer's guidelines, the analytical method, and/or the requirements of special contracts determine the frequency of calibration and the concentration of calibration standards, whichever is most applicable. The following are very general guidelines and are not meant to be all-inclusive. Detailed calibration procedures are specified in the SOP for each method performed.

<u>Gas Chromatography/Mass Spectroscopy (GC/MS)</u>: Each day prior to analysis of samples, all GC/MS instruments are tuned with 4-bromofluorobenzene (BFB) for VOCs and decafluorotriphenylphosphine (DFTPP) for SVOCs in accordance with the tuning criteria specified in the applicable methods. Samples are not analyzed until the method-specific tuning requirements have been met.

After the tuning criteria are met, the instrument is then calibrated for all target analytes and an initial multipoint calibration curve established. Alternatively, the previous calibration curve may be used if validated by a calibration verification (CV) standard. All target analytes are represented in the calibration and certain key target analytes referred to as system performance calibration compounds (SPCCs) and calibration check compounds (CCCs) are used for curve acceptance determination. For the initial calibration to be deemed acceptable, the SPCCs and CCCs must meet established acceptance criteria and must be re-evaluated and meet the acceptance criteria, at a minimum, every twelve (12) hours thereafter.

Non-GC/MS Chromatography: The field of chromatography involves a variety of instrumentation and detectors. While calibration standards and control criteria vary depending upon the type of system and analytical methodology required for a specific analysis, the general principles of calibration apply uniformly. Each chromatographic system is calibrated prior to sample analysis. An initial multipoint calibration curve is generated using all target analytes. All target analytes must meet the acceptance criteria for the calibration to be deemed acceptable. The continued validity of the initial multipoint calibration is verified every 12 hours using a calibration verification (CV) standard containing all target analytes. If the CV fails to meet the acceptance criteria, the system is re-calibrated and all samples analyzed since the last acceptable CV must be re-analyzed.

Inductively Coupled Plasma Emission Spectroscopy: Initial calibration consists of a calibration blank (CB) plus one calibration standard. The calibration is verified by the re-analysis of the standard and initial calibration verification (ICV) standard. If the standard and the ICV fail to meet the acceptance criteria, the initial calibration is considered invalid and is re-performed.

Continuing calibration verification (CCV) consists of a mid-concentration standard plus a calibration blank (CB) analyzed every 10 samples and at the end of the sequence. If the CCV and/or CB fail to meet the acceptance criteria, the instrument must be re-calibrated and all samples analyzed since the previous acceptable CCV and/or CB must be re-analyzed.

<u>ICP/MS Spectroscopy</u>: Each day prior to the analysis of samples, all ICP/MS instruments undergo mass calibration and resolution checks prior to initial calibration. Initial calibration consists of a calibration blank (CB) and at least one calibration standard. The calibration is verified by the reanalysis of the standard and initial calibration verification (ICV) standards. If the standard and the ICV fail to meet the acceptance criteria, the initial calibration is considered invalid and is reperformed.

Continuing calibration verification (CCV) consists of a mid-concentration standard plus a calibration blank (CB) analyzed every 10 samples and at the end of the sequence. If the CCV and/or CB fail to meet the acceptance criteria, the instrument must be re-calibrated and all samples analyzed since the previous acceptable CCV and/or CB must be re-analyzed.

<u>Flame and Graphite Furnace Atomic Absorption Spectroscopy</u>: Initial calibration consists of a calibration blank plus a low, medium, and high calibration standard. Continuing calibration verification (CCV) consists of midpoint calibration standard plus a calibration blank (CB) analyzed every 10 samples and at the end of the sequence. If the CCV and/or CB fail to meet the acceptance criteria, the instrument must be re-calibrated and all samples analyzed since the previous acceptable CCV and/or CB must be re-analyzed. If the calibration blanks contain target analyte concentrations exceeding the acceptance limits, the cause must be determined and corrected.

<u>General Inorganic Analyses</u>: General inorganic (non-metal) analyses involve a variety of instrumental and wet chemistry techniques. While calibration procedures vary depending on the type of instrumentation and methodology, the general principles of calibration apply universally. Each system or method is initially calibrated using standards prior to analyses being conducted with continual verification that the calibration remains acceptable throughout analytical processing. If continual calibration verification fails to meet the acceptance criteria, the instrument must be recalibrated and all samples analyzed since the previous acceptable CCV must be re-analyzed.

o) Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

These procedures may be found in SOP-T015 (Correction/Prevention of Errors in Test Records) and SOP-T022 (Corrective/Preventive Actions).

p) The laboratory management arrangements for permitting exceptions and departures from documented policies and procedures or from standard specifications;

Calscience's SOPs are in substantial conformity with their corresponding published method references. Departure from approved SOPs shall be approved if necessary or appropriate due to the nature or composition of the sample or otherwise based on the reasonable judgment of Calscience's Laboratory Director, Technical Director, or QA Manager. Departures shall be made on a case-by-case basis consistent with recognized standards of the industry. In no case shall departures be approved without written communication between Calscience and the affected client.

q) Procedures for dealing with complaints;

Procedures for dealing with complaints may be found in SOP-T018, Handling of Inquiries and Complaints.

r) Procedures for protecting confidentiality (including national security concerns) and proprietary rights;

Calscience is sensitive to the fact that much of the analytical work performed for clientele may be subject to litigatory processes. Calscience, therefore, holds all information in strict confidence with laboratory release only to the client or designee. Information released to entities other than the client is performed only upon written, facsimile or e-mail request from the client.

Due to the investigative nature of most site assessments, analytical information may become available to regulatory agencies or other evaluating entities during site assessment of the laboratory for the specific purpose of attaining laboratory certifications, accreditations, or evaluation of laboratory qualification for future work. During these occurrences, the laboratory will make its best effort to maintain the confidence of client specific information.

s) Procedures for audits and data review:

Calscience participates in a wide variety of system and performance audits conducted by numerous federal and state agencies, as well as through its major clientele. These audits are conducted to verify that analytical data produced conforms to industry standards on a routine basis.

A System Audit is a qualitative evaluation of the measurement systems utilized at Calscience, specifically, that Calscience has, in place, the necessary facilities, staff, procedures, equipment, and instrumentation to generate acceptable data. This type of audit typically involves an on-site inspection of the laboratory facility, operations, and interview of personnel by the auditing agency.

A Performance Audit verifies the ability of Calscience to correctly identify and quantitate compounds in blind check samples. This type of audit normally is conducted by the auditing agency through laboratory participation in round robin Performance Evaluation (PE) programs. Examples of current PE program involvement include those offered by commercial suppliers like ERA (WS/WP/SOIL and DMR-QA), or other inter-laboratory studies not required for certification but done to ensure laboratory performance, as well as programs administered by major industry.

Outliers in required PE samples will be investigated and corrective actions documented using the Corrective/Preventive Action Record.

In addition to performance and system audits conducted by auditing agencies or clients, Calscience's QA Manager in association with the Laboratory Director regularly generates quarterly QA Reports.

A reporting system is a valuable tool for measuring the overall effectiveness of Calscience's QA program. It serves as an instrument for evaluating the program's design, identification of problems and trends, and planning for future needs.

The Quarterly QA Reports normally addresses the following information:

- ⇒ Laboratory certifications and approvals;
- ⇒ System and performance audits:
- ⇒ Performance evaluation studies:
- ⇒ LIMS
- ⇒ Performance on major contracts; and
- ⇒ Miscellaneous issues.

The QA goals for the following year will be included in the last Quarterly QA Report of every year.

Should the result of any audit detect a significant error, which has been identified to adversely affect released data, the situation shall be thoroughly investigated. Corrective measures shall be enacted to include system re-evaluation, the determined affect on released data and client notification, as necessary. These measures shall be documented using the Corrective/Preventive Action Record.

t) Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training;

Quality control begins prior to sample(s) receipt at the laboratory. The selection of well qualified personnel, based upon education and/or experience is the first step in successful laboratory management. A thorough screening of job applicants and selection of the best candidate to fulfill a well-defined need is as important an aspect of a successful QA/QC program as a careful review of analytical data.

Employee training and approval procedures used at Calscience are specified in SOP-T010, "Employee Training", and includes but is not limited to the following:

- ⇒ A thorough understanding of the applicable regulatory method and Calscience SOP;
- A review of Calscience's QA Program Manual and thorough understanding of the specifics contained therein that are directly related to the analysis to be performed;
- ⇒ Instruction by the applicable Group Leader on all aspects of the analytical procedure;
- ⇒ Performance of analyses under supervision of experienced laboratory personnel, which shall include analysis of blind QC check samples, when deemed appropriate;
- ⇒ Participation in in-house seminars on analytical methodologies and procedures;
- ⇒ Participation in job related seminars outside of the laboratory; and
- ⇒ Participation in conventions and meetings, i.e., ACS, etc.
- Ethics policy statement developed by the laboratory and processes/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical, or illegal actions;

A vital part of Calscience Environmental Laboratories' analytical laboratory services is their Laboratory Ethics Training Program. An effective program starts with an Ethics Policy Statement that is supported by all staff, and is reinforced with initial and ongoing ethics training.

"It shall be the policy of Calscience to conduct all business with integrity and in an ethical manner. It is a basic and expected responsibility of each staff member and manager to hold to the highest ethical standard of professional conduct in the performance of all duties."

A proactive ethics training program is the most effective means of deterring and detecting improper, unethical, or illegal actions in the laboratory. There are four facets to the program: (1) clearly define improper, unethical, and illegal actions; (2) outline elements of prevention and detection programs for improper, unethical, or illegal actions; and (3) identify examples of inappropriate (i.e., potentially fraudulent) laboratory practices; (4) Annual Ethics Training

Definition of Improper, Unethical, and Illegal Actions

Improper actions are defined as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional.

Unethical or illegal actions are defined as the deliberate falsification of analytical or quality assurance results, where failed method or contractual requirements are made to appear acceptable.

Prevention of laboratory improper, unethical, or illegal actions begins with a zero-tolerance philosophy established by management. Improper, unethical, or illegal actions are detected through the implementation of oversight protocols.

Prevention and Detection Program for Improper, Unethical, or Illegal Actions

Calscience management has implemented a variety of proactive measures to promote prevention and detection of improper, unethical, or illegal activities. The following components constitute the basic program:

- ⇒ Data Integrity Standard Operating Procedure (SOP) T065
- ⇒ Data Integrity Documentation Procedures
- ⇒ An Ethics and Data Integrity Agreement that is read and signed by all personnel;
- ⇒ Initial and annual ethics training;
- ⇒ Internal audits:
- ⇒ Inclusion of anti-fraud language in subcontracts;
- ⇒ Analyst notation and sign-off on manual integration changes to data;
- ⇒ Active use of electronic audit functions when they are available in the instrument software; and

⇒ A "no-fault" policy that encourages laboratory personnel to come forward and report fraudulent activities.

A proactive, "beyond the basics" approach to the prevention of improper, unethical, or illegal actions are a necessary part of laboratory management. As such, in addition to the requirements above, Calscience has a designated ombudsman (data integrity officer) to whom laboratory personnel can report improper, unethical, or illegal practices, or provide routine communication of training, lectures, and changes in policy intended to reduce improper, unethical, or illegal actions.

Examples of Improper, Unethical, or Illegal Practices

Documentation that clearly shows how all analytical values were obtained are maintained by Calscience and supplied to the data user as needed. To avoid miscommunication, Calscience clearly documents all errors, mistakes, and basis for manual integrations within the project file and case narrative as applicable. Notification is also made to the appropriate supervisor so that appropriate corrective actions can be initiated. Gross deviations from specified procedures are investigated for potential improper, unethical, or illegal actions, and findings of fraud are fully investigated by senior management. Examples of improper, unethical, or illegal practices are identified below:

- ⇒ Improper use of manual integrations to meet calibration or method QC criteria (for example, peak shaving or peak enhancement are considered improper, unethical, or illegal actions if performed solely to meet QC requirements);
- Intentional misrepresentation of the date or time of analysis (for example, intentionally resetting a computer system's or instrument's date and/or time to make it appear that a time/date requirement was met);
- ⇒ Falsification of results to meet method requirements;
- ⇒ Reporting of results without analyses to support (i.e., dry-labbing);
- ⇒ Selective exclusion of data to meet QC criteria (for example, initial calibration points dropped without technical or statistical justification);
- ⇒ Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported, or QC control limits presented within QAPP that are not indicative of historical laboratory performance or used for batch control;
- ⇒ Notation of matrix inference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (for example, method blanks or laboratory control samples);
- ⇒ Unwarranted manipulation of computer software (for example, improper background subtraction to meet ion abundance criteria for GC/MS tuning, chromatographic baseline manipulations);
- ⇒ Improper alteration of analytical conditions (for example, modifying EM voltage, changing GC temperature program to shorter analytical run time) from standard analysis to sample analysis;
- ⇒ Reporting of results from the analysis of one sample for those of another.

v) Reference to procedures for reporting analytical results;

Standard operating procedures pertaining to the reporting of results are available to all laboratory personnel. They are: SOP-T009, Significant Figures, Rounding, and Reporting of Results; SOP-T025, Reporting of Tentatively Identified Compounds (TICs); and T-026, Reporting of Data Qualifiers.

All analytical data generated within Calscience is thoroughly checked for accuracy and completeness. The data validation process consists of data generation, reduction, and four levels of review as described below.

The analyst generating the analytical data has the primary responsibility for its correctness and completeness. All data is generated and reduced following protocols specified in the appropriate SOPs. Each analyst reviews the quality of his or her work based upon an established set of guidelines specified in the SOPs or as specified by project requirements. The analyst reviews the data package to ensure that:

- ⇒ Holding times have not been exceeded;
- ⇒ Sample preparation information is correct and complete;
- ⇒ Analysis information is correct and complete;
- ⇒ The appropriate procedures were employed;
- ⇒ Analytical results are correct and complete;
- All associated QC is within established control limits and, if not, out-of-control forms are completed thoroughly explaining the cause and corrective action taken;
- ⇒ Any special sample preparation and analytical requirements have been met; and
- Documentation is complete, i.e., all anomalies in the preparation and analysis have been documented; out-of-control forms, if required, are complete, etc.

The data reduction and validation steps are documented, signed, and dated by the analyst on the QC Review coversheet accompanying each data package. This initial review step, performed by the analyst, is designated as primary review. The analyst then forwards the data package to his or her Group Leader, or designated data reviewer, who performs a secondary review. Secondary reviews consist of an independent check equivalent to that of the primary review and are designed to ensure that:

- ⇒ Calibration data is scientifically sound, appropriate to the method, and completely documented;
- ⇒ QC data is within established guidelines or reported with appropriate clarification/qualification;
- □ Qualitative identification of sample components is correct;
- ⇒ Quantitative results are correct:
- ⇒ Documentation is complete and any anomalies properly addressed and documented;
- ⇒ The data is ready for incorporation into the final report package; and
- ⇒ The data package is complete and ready for archiving.

A significant component of the secondary review is the documentation of any errors that have been identified and corrected during the review process. Calscience believes that the data package that is submitted for a secondary review should be free from errors. Errors that are discovered are documented and formally transmitted to the appropriate Group Leader. The cause of the errors are then addressed by additional training or clarification of procedures (SOP revisions) to ensure that similar errors do not recur and high quality data will be generated.

Signature of Data Reviewer and the date of review document the completion of secondary reviews on the QC Review coversheet. These constitute approval for data release and generation of analytical report.

During both of the QC review processes, 100% of the raw data associated with the entire project is available to the reviewer. Data packages are checked back to the raw data as deemed necessary by the reviewer.

Following draft report generation, the report is reviewed by the Project Manager to ensure that the data set and quality control data is complete and meets the specific requirements of the project. When available, the data is also evaluated against historical site information. Once all requested analytical work has been verified as complete, a final report is generated and signed by the Project Manager.

Following approval for release by the Project Manager, the Quality Assurance Manager or Designee to ensure that the analytical and quality control data is correct performs a final review. The Quality Assurance Manager may review 10% of the project files back to the raw data as an additional check.

A variety of reporting formats, from Portable Document File (PDF), normal typed reports to computerized data tables to complex reports discussing regulatory issues are available. In general, Calscience reports contain the following information.

Analytical Data

Analytical data is reported by sample identification (both client and laboratory) and test. Pertinent information including date(s) sampled, received, prepared, and analyzed; any required data qualifiers are included on each results page. The reporting limit for each method analyte is also listed. Additional data may include Method Detection Limits (MDLs).

QC Data

A QC Summary is provided with each final report. Unless otherwise specified in a QAPP or requested by the client, QC Summaries include results for method blanks, matrix spikes, matrix spike duplicates, and surrogate spikes. Laboratory control sample and method blank surrogates are routinely included if matrix interference results in a QC outlier. The effective control limits for the reported QC values are also provided on the QC Summary as well as explanations for any QC outliers. Case Narratives may be included as appropriate.

As required for the project, data reports from "results only" through "full CLP" will be generated and provided. Included in this range are reports for the major DoD programs including NFESC, AFCEE, and USACE.

Methodology

References for the preparative and analytical methodology employed is included on all preliminary or final analytical reports.

Signatory

Final reports are ready for release to the client following review and approval by the Project Manager, as evidenced by his/her signature on the final report cover page.

Preliminary Data

Upon client request, preliminary data shall be released prior to completion of a full QC review. Preliminary data is subject to change pending QC review and, therefore, shall be clearly marked as "Preliminary, QC Pending" and not include a signature of approval. This qualification is provided as notification to the client that the data review process has not been completed yet and that the data is subject to possible modification resulting therefrom.

Revised Data

Analytical reports that have been revised for any reason from the original sent report shall be noted as being revised with a report note, case narrative or indication as to the revision.

Formatting

At a minimum, an analytical report shall consist of the Report Cover Page, Analytical Results, QA/QC Data (Default), Footnotes/Comments Page, Sample Receipt Form and COC. Paginated reports shall be employed for all reports unless used for non-NELAP analysis.

w) A Table of Contents and applicable lists of references and glossaries, and appendices.

5.3 Audits

5.3.1 Internal Audits

The laboratory arranges streamlined quarterly and comprehensive annual internal audits to verify that its operations continue to comply with the requirements of the laboratory's said quality system. The quality assurance officer plans and organizes audits as required by a predetermined schedule and requested by management. Trained and qualified personnel, who are wherever resources permit, independent of the activity to be audited, carry out such audits. Personnel do not audit their own activities except when it can be demonstrated that an effective audit will be carried out. Where the audit findings cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory takes immediate corrective action and immediately notifies, in writing, any client whose work was involved.

The outcome of internal audits is included in the applicable quarterly report to management. The QA Manager is responsible for maintaining these reports.

5.3.2 Managerial Review

Calscience management conducts an annual review of its quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. This review takes account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of inter-laboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions, and other relevant factors. The laboratory shall have a procedure for review by management, and maintain records of review findings and actions.

5.3.3 Audit Review

All audit and review findings and any corrective actions that arise from them are documented. The laboratory management ensures that these actions are discharged within the agreed time frame as indicated in the quality manual and/or SOPs.

5.3.4 Performance Audits

In addition to periodic audits, the laboratory ensures the quality of results provided to clients by implementing checks to monitor the quality of the laboratory's analytical activities. Examples of such checks are:

- a) Internal quality control procedures using statistical techniques (see Section 5.4 below);
- b) Participation in proficiency testing or other interlaboratory comparisons;
- c) Use of certified reference materials and/or in-house quality control using secondary reference materials as specified in Calscience QSM Section 5.4;
- d) Replicate testing using the same or different test methods;

- e) Re-testing of retained samples;
- e) Correlation of results for different but related analysis of a sample (for example, total phosphorus should be greater than or equal to orthophosphate).

5.3.5 Corrective / Preventive Actions

- a) In addition to providing acceptance criteria and specific protocols for corrective/preventive actions in SOP-T022, the laboratory implements general procedures to be followed to determine when departures from documented policies, procedures and quality control have occurred. These procedures include but are not limited to the following:
 - 1) Identify the individual(s) responsible for assessing each QC data type;
 - 2) Identify the individual(s) responsible for initiating and/or recommending corrective/preventive actions;
 - Define how the analyst shall treat a data set if the associated QC measurements are unacceptable;
 - Specify how out-of-control situations and subsequent corrective actions are to be documented;
 and
 - 5) Specify procedures for management (including the QA officer) to review corrective/preventive action reports.
- b) To the extent possible, sample results are reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data are to be reported, all samples associated with the failed quality control measure are reported with the appropriate data qualifier(s).

5.4 Essential Quality Control Procedures

These general quality control principles apply, where applicable, to all testing at Calscience. The manner in which each is implemented is dependent on the types of tests performed by the laboratory and is further described in Appendix D and in SOP-T020 (Internal Quality Control Checks. The standards for any given test type assures that the applicable principles are addressed:

- a) All laboratories have detailed written protocols in place to monitor the following quality controls:
 - 1) Positive and negative controls (blanks, spikes, reference toxicants, etc.) to monitor tests;
 - 2) Tests to define the variability and/or repeatability of the laboratory results such as replicates;
 - 3) Measures to assure the accuracy of the test method including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures:
 - 4) Measures to evaluate test method capability, such as detection limits and quantitation limits or range of applicability such as linearity;
 - 5) Selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;
 - 6) Selection and use of reagents and standards of appropriate quality;

- 7) Measures to assure the selectivity of the test for its intended purpose; and
- 8) Measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the test method, such as temperature, humidity, light, or specific instrument conditions.
- b) All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance criteria are used to determine the usability of the data. (See Appendix D.)
- c) The laboratory has procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See Calscience QSM Section 11.2, Sample Acceptance Policy.)
- d) The quality control protocols specified in the method manual (Calscience QSM Section 10.1.2) is followed. Calscience ensures that the essential standards outlined in NELAC 5, Appendix D, or mandated methods or regulations (whichever are more stringent) are incorporated into the method manuals. When it is not apparent which is more stringent the QC in the mandated method or regulations is to be followed.

The essential quality control measures for testing are found in Appendix D.

6.0 PERSONNEL

6.1 General Requirements for Laboratory Staff

Calscience's testing departments have a sufficient level of personnel with the necessary education, training, technical knowledge and experience to perform the assigned functions.

All personnel are responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures and records management.

6.2 Laboratory Management Responsibilities

In addition to Calscience QSM Section 4.2.d, the laboratory management:

- a) Defines the minimum level of qualification, experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills such as using a balance and quantitative techniques, are considered.
- b) Ensures that all technical laboratory staff members demonstrate capability in the activities for which they are responsible. Such demonstration is documented (See Appendix C).
 - Note: In departments with specialized "work cells" (a well-defined group of analysts that together perform the method analysis), the group as a unit meets the above criteria and this demonstration is fully documented.
- c) Ensures that the training of each member of the technical staff is kept up-to-date (on-going) by the following:

- Keeping evidence on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation that relates to his/her job responsibilities.
- 2) Documenting training courses or workshops on specific equipment, analytical techniques, or laboratory procedures.
- 3) Documenting employee attendance at training courses on ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions. Keeping on file evidence that demonstrates that each employee has read, acknowledges, and understands their personal ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions.
- 4) Maintains up-to-date analyst training records that contain a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or standard operating procedure as defined by the laboratory document control system, Calscience QSM Section 5.2.d) and documentation of continued proficiency by at least one of the following once per year:
 - i. Acceptable performance of a blind sample (single blind to the analyst);
 - ii. Another demonstration of capability;
 - iii. Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624, or 5035/8260) would only require documentation for one of the test methods;
 - iv. At least four consecutive laboratory control samples with acceptable levels of precision and accuracy;
 - v. If i-iv cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- d) Documents all analytical and operational activities of the laboratory;
- e) Supervises all personnel employed by the laboratory;
- f) Ensures that all sample acceptance criteria (Calscience QSM Section 11.0) are verified and that samples are logged into the sample tracking system and properly labeled and stored.
- g) Documents the quality of all data reported by the laboratory.
- h) Develops a proactive program for the prevention and detection of improper, unethical, or illegal actions. Components of this program could include: internal proficiency testing (single and double blind); post-analysis electronic and magnetic tape audits; effective reward program to improve employee vigilance and co-monitoring; and separate SOPs identifying appropriate and inappropriate laboratory and instrument manipulation practices.

6.3 Records

Records on the relevant qualifications, training, skills and experience of the technical personnel are maintained by the laboratory (see Calscience QSM Section 6.2.c), including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in Calscience QSM Section 10.2.1 for chemical testing.

7.0 PHYSICAL FACILITIES – ACCOMMODATION AND ENVIRONMENT

7.1 Environment

- a) Laboratory accommodations, test areas, energy sources, lighting, heating and ventilation are such that they facilitate proper performance of tests.
- b) The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of the measurements. Particular care shall be taken when such activities are undertaken at sites other than the permanent laboratory premises.
- c) The laboratory shall provide for the effective monitoring, control and recording of environmental conditions as appropriate. Such environmental conditions may include biological sterility, dust, electromagnetic interference, humidity, main voltage, temperature, and sound and vibration levels.
- d) In instances where monitoring or control of any of the above-mentioned items is specified in a test method or by regulation, the laboratory meets and documents adherence to the laboratory facility requirements.

7.2 Work Areas

- a) There is effective separation between neighboring areas when the activities therein are incompatible including volatile organic chemicals handling areas.
- b) Access to and use of all areas affecting the quality of these activities are defined and controlled.
- c) Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.
- d) Workspaces are available to ensure an unencumbered work area. Work areas include:
 - 1) Access and entryways to the laboratory;
 - 2) Sample receipt areas:
 - 3) Sample storage areas;
 - 4) Chemical and waste storage areas; and
 - 5) Data handling and storage areas.

8.0 EQUIPMENT AND REFERENCE MATERIALS

- a) Calscience is furnished with all items of equipment (including reference materials) required for the correct performance of tests for which accreditation is maintained. Note that Calscience does not use equipment outside its permanent control.
- b) All equipment is properly maintained, inspected, and cleaned. Maintenance procedures are documented.
- c) Any equipment item that has been subjected to overloading or mishandling, or that gives suspect results, or has been shown by verification or otherwise to be defective, is taken out of service, clearly identified and wherever possible stored at a specified place until it has been repaired and shown by

calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations or tests.

- d) When appropriate, each item of equipment, including reference materials, is labeled, marked, or otherwise identified to indicate its calibration status.
- e) Records are maintained of each major item of equipment and all reference materials significant to the tests performed. These records include documentation on all routine and non-routine maintenance activities in assigned log books and reference material verifications.

The records include:

- 1) The name of the item of equipment;
- 2) The manufacturer's name, type identification, and serial number or other unique identification;
- 3) Date received and date placed in service (if available);
- 4) Current location, where appropriate;
- 5) If available, condition when received (e.g., new, used, reconditioned);
- 6) Copy of the manufacturer's instructions, where available;
- 7) Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;
- 8) Details of maintenance carried out to date and planned for the future; and
- 9) History of any damage, malfunction, modification or repair.

9.0 MEASUREMENT TRACEABILITY AND CALIBRATION

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before being put into service and on a continuing basis. The laboratory has an established program for the calibration and verification of its measuring and test equipment. This includes balances, thermometers and control standards.

9.2 Traceability of Calibration

- a) The overall program of calibration and/or verification and validation of equipment is designed and operated so as to ensure that measurements made by the laboratory are traceable to national standards of measurement.
- b) Calibration certificates indicate the traceability to national standards of measurement and provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory maintains records of all such certification in the QA office.

c) Where traceability to national standards of measurement is not applicable, the laboratory provides satisfactory evidence of correlation of results, for example, by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.

9.3 Reference Standards

- a) Reference standards of measurement held by the laboratory (such as Class S or equivalent weights, or traceable thermometers) are used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards has not been invalidated. A body that can provide traceability calibrates reference standards of measurement. Where possible, this traceability is to a national standard of measurement.
- b) There is a program of calibration and verification for reference standards.
 - Two weeks prior to their date of calibration expiration, individual thermometers are removed from service and replaced by newly calibrated units from the supplier.
 - ii. Calscience keeps two sets of Class S weights on hand for use in the laboratory. One set is used for daily calibration checks, and the second set is kept for back up use should the first set be damaged, lost or otherwise compromised. The second set of weights is also place in service when the daily use set is shipped off site for recalibration.
 - iii. Analytical balances are serviced and calibrated on a routine, annual schedule.
- c) Where relevant, reference standards and measuring and testing equipment are subjected to inservice checks between calibrations and verifications. Reference materials are traceable. Where possible, traceability is to national or international standards of measurement, or to national or international standard reference materials.

9.4 Calibration

Calibration requirements are divided into two parts: (1) requirements for analytical support equipment, and (2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

9.4.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume.

- a) All support equipment is maintained in proper working order. The records of all repair and maintenance activities, including service calls is kept.
- b) All support equipment is calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration are within the specifications required of the application for which this equipment is used or:
 - 1) The item is removed from service until repaired; or
 - The laboratory maintains records of established correction factors to correct all measurements.

- c) Raw data records are retained to document equipment performance.
- d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths are checked in the expected use range, with NIST traceable references where available. The acceptability for use or continued use is according to the needs of the analysis or application for which the equipment is being used.
- e) Mechanical volumetric dispensing devices including burettes (except Class A glassware) is checked for accuracy on at least a quarterly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware, comes with a certificate attesting to established accuracy or the accuracy is initially demonstrated and documented by the laboratory.

9.4.2 Instrument Calibration

This manual specifies the essential elements that define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data are of known quality and be appropriate for a given regulation or decision. This manual does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory demonstrates that such requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are to be followed.

Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration.

9.4.2.1 Initial Instrument Calibrations

The following items are essential elements of initial instrument calibration:

- a) The details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics are included or referenced in the test method SOP. When initial instrument calibration procedures are referenced in the test method, the referenced material is retained by the laboratory and is available for review.
- b) Sufficient raw data records are retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, analyst's initials or signature; concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration.
- c) Sample results are quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.
- d) All initial instrument calibrations is verified with a standard obtained from a second manufacturer or lot. Traceability shall be to a national standard, when available.
- e) Criteria for the acceptance of an initial instrument calibration is established, e.g., correlation coefficient or relative percent difference. The criteria used is appropriate to the calibration technique employed.

- f) Results of samples not bracketed by initial calibration standards (within calibration range) are reported as having less certainty, e.g., defined qualifiers or flags or explained in the case narrative. As determined by the method, the lowest calibration standard is at or above the detection limit.
- g) If the initial instrument calibration results are outside established acceptance criteria, corrective actions are performed. Data associated with an unacceptable initial instrument calibration is not reported.
- h) Calibration standards include concentrations at or below the regulatory limit/decision level, if the laboratory knows these limits/levels, unless these concentrations are below the laboratory's demonstrated detection limits (See Calscience QSM Section Appendix D.1.4 Detection Limits).
- i) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, not including blanks or a zero standard. The laboratory's standard operating procedure defines the number of points for establishing the initial instrument calibration.

9.4.2.2 Continuing Instrument Calibration Verification

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration is verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

- a) The details of the continuing instrument calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.
- b) A continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch. The concentrations of the calibration verification shall be varied within the established calibration range. If an internal standard is used, only one continuing instrument calibration verification must be analyzed per analytical batch.
- c) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations. Continuing calibration verification records must explicitly connect the continuing verification data to the initial instrument calibration.
- d) Criteria for the acceptance of a continuing instrument calibration verification must be established, e.g., relative percent difference.
- e) If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second (consecutive and immediate) calibration verification within acceptance criteria, then the laboratory shall demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new instrument calibration must be performed. If the laboratory has not demonstrated acceptable performance, sample analyses shall not occur until a new initial calibration curve is established and verified.

As an exception, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

i. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias and there are associated samples that are non-detects, then those non-detects may

be reported. Otherwise the samples affected by the unacceptable calibration verification are reanalyzed after a new calibration curve has been established, evaluated and accepted.

ii. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification are reanalyzed after a new calibration curve has been established, evaluated and accepted.

10.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

10.1 Methods Documentation

- a) The laboratory has documented instructions on the use and operation of all relevant equipment, on the handling and preparation of samples and for calibration and/or testing, where the absence of such instructions could jeopardize the calibrations or tests.
- b) All instructions, standards, manuals, and reference data relevant to the work of the laboratory are maintained up-to-date and be readily available to the staff.

10.1.1 Standard Operating Procedures (SOPs)

Calscience maintains standard operating procedures that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

- a) These documents, for example, may be equipment manuals provided by the manufacturer or internally written documents.
- b) The test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the methods manual. (See 10.1.2.)
- c) Copies of all SOPs are accessible to all personnel.
- d) The SOPs are organized.
- e) Each SOP clearly indicates the effective date of the document, the revision number and the signatures of the approving authorities.

10.1.2 Laboratory Method Manual(s)

- a) The laboratory has and maintains an in-house methods manual for each accredited analyte or test method.
- b) This manual may consist of copies of published or referenced test methods or standard operating procedures that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described. Each test method includes or references where applicable:
 - 1) Identification of the test method;
 - 2) Applicable matrix or matrices;
 - 3) Detection limit;
 - 4) Scope and application, including components to be analyzed;
 - 5) Summary of the test method;

- 6) Definitions:
- 7) Interferences;
- 8) Safety;
- 9) Equipment and supplies:
- 10) Reagents and standards;
- 11) Sample collection, preservation, shipment, and storage;
- 12) Quality control;
- 13) Calibration and standardization;
- 14) Procedure:
- 15) Calculations:
- 16) Method performance;
- 17) Pollution prevention;
- 18) Data assessment and acceptance criteria for quality control measures;
- 19) Corrective actions for out-of-control data;
- 20) Contingencies for handling out-of-control or unacceptable data;
- 21) Waste management;
- 22) References; and
- 23) Any tables, diagrams, flowcharts, and validation data.

Laboratory procedures other than preparative or analytical procedure may use a shortened format as outlined in SOP T001.

10.2 Test Methods

The laboratory uses appropriate test methods and procedures for all tests and related activities within its responsibility (including, as applicable, sample collection, sample handling, transport and storage, sample preparation and sample analysis). The method and procedures shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations or tests concerned.

- a) When the use of specific test methods for a sample analysis is mandated or requested, only those methods are used.
- b) Where test methods are employed that are not required, as in the Performance Based Measurement System approach, the methods are fully documented and validated (see Calscience QSM Section 10.2.1 and Appendix C), and are available to the client and other recipients of the relevant reports.

10.2.1 Demonstration of Capability

- a) Prior to acceptance and institution of any test method, satisfactory demonstration of method capability is required. (See Calscience QSM Section Appendix C and 6.2.b.) This demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (sample of a matrix is which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids and air. In addition, for analytes that do not lend themselves to spiking, the demonstration of capability may be performed using quality control samples.
- b) Continuing demonstration of method performance, as per the quality control requirements in Appendix D (such as laboratory control samples) is required.
- c) In cases where Calscience analyzes samples using a test method that has been in use by the laboratory before July 1999, and there have been no significant changes in instrument type, personnel or test method, the continuing demonstration of method performance and the analyst's documentation of continued proficiency shall be acceptable. The laboratory shall have records on file to demonstrate that an initial demonstration of capability is not required.

- d) In all cases, the appropriate forms, such as the Certification Statement (Appendix C), is completed and retained by the laboratory to be made available upon request. The laboratory retains all associated supporting data necessary to reproduce the analytical results summarized in the Certification Statement. (See Appendix C for an example of a Certification Statement.)
- e) Demonstration of capability is completed each time there is a significant change in instrument type, personnel, or test method.
- f) In departments with specialized "work cell(s)" (a group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit must meet the above criteria and this demonstration of capability is fully documented.
- g) When a work cell is employed, and the members of the cell change, the new employee(s) must work with an experienced analyst in that area of the work cell where they are employed. This new work cell must demonstrate acceptable performance through acceptable continuing performance checks (appropriate sections of Appendix D, such as laboratory control samples). Such performance is documented and the four preparation batches following the change in personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. In addition, if the entire work cell is changed or replaced, the new work cell must perform the demonstration of capability (Appendix C).
- h) Performance of the work cell is linked to the training records of the individual members of the work cell (See Calscience QSM Section 6.2).

10.3 Sample Aliquots

Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples. Reference SOP M230 "Homogenization and Compositing of Solid, Soil and Sediment sample" for further guidance.

10.4 Data Verification

Calculations and data transfers are subject to appropriate checks.

- a) The laboratory has Standard Operating Procedures that ensure that the reported data are free from transcription and calculation errors.
- b) The laboratory has Standard Operating Procedures that ensure that all quality control measures are reviewed, and evaluated before data are reported.
- The laboratory has Standard Operating Procedures that address manual calculations including manual integrations.

10.5 Documentation and Labeling of Standards and Reagents

Documented procedures exist for the purchase, receipt and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory retains records for all standards, reagents and media including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material is not used, unless the laboratory verifies its suitability for testing use.

- b) Original containers (such as those provided by the manufacturer or vendor) are labeled with an expiration date.
- c) Records are maintained on reagent and standard preparation. These records indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.
- d) All containers of prepared reagents and standards bear a unique identifier and expiration date and are linked to the documentation requirements in Calscience QSM Section 10.5.c above.

10.6 Computers and Electronic Data Related Requirements

Where computers, automated equipment, or microprocessors are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data, Calscience ensures that:

- a) All requirements of the NELAC Standard (i.e., Chapter 5 of NELAC) are met;
- b) Computer software is tested and documented to be adequate for use, e.g., internal audits, personnel training, focus point of QA and QC;
- Procedures are established and implemented for protecting the integrity of data. Such procedures include, but are not limited to, integrity of data entry or capture, data storage, data transmission and data processing;
- d) Computer and automated equipment are maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data: and
- e) It establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

11.0 SAMPLE HANDLING, SAMPLE ACCEPTANCE POLICY AND SAMPLE RECEIPT

While Calscience does not have control of field sampling activities, the following are essential to ensure the validity of the laboratory's data.

11.1 Sample Tracking

- a) The laboratory has a documented system for uniquely identifying the items to be tested, to ensure that there can be no confusion regarding the identity of such items at any time. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory assigns a unique identification (ID) code to each sample container received in the laboratory. (The use of container shape, size, or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.)
- b) This laboratory code is maintained as an unequivocal link with the unique field ID code assigned each container.
- c) The laboratory ID code is placed on the sample container as a durable label.

- d) The laboratory ID code is entered into the laboratory records (see Calscience QSM Section 11.3.d) and is the link that associates the sample with related laboratory activities such as sample preparation or calibration.
- e) In cases where the sample collector and analyst are the same individual or the laboratory pre-assigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

11.2 Sample Acceptance Policy

The laboratory has a written sample acceptance policy that clearly outlines the circumstances under which samples are accepted or rejected. Data from any samples that do not meet the following criteria are flagged in an unambiguous manner, and the nature of the variation is clearly defined. The sample acceptance policy is available to sample collection personnel and includes, but is not limited to, the following areas of concern:

- a) Proper, full, and complete documentation, that includes sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;
- b) Proper sample labeling that includes a unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;
- c) Use of appropriate sample containers;
- d) Adherence to specified holding times;
- e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests; and
- f) Procedures to be used when samples show signs of damage, contamination or inadequate preservation.
- g) Samples are NOT accepted if classified as extremely hazardous, reference section 5.2 k for examples.

11.3 Sample Receipt Protocols

- upon receipt, the condition of the sample, including any abnormalities or departures from standard condition as prescribed in the relevant test method, is recorded. All items specified in Calscience QSM Section 11.2 above are checked.
 - 1) All samples that require cold temperature preservation are considered acceptable if the arrival temperature is within 2°C of the required temperature or the method-specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet these criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.
 - The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or free chlorine, prior to or during sample preparation or analysis.

With the exception of residual chlorine measurements in aquatic toxicity samples, certain measurements, such a pH, are performed and recorded just prior to analysis.

- b) The results of all checks are recorded.
- c) When there is any doubt as to the item's suitability for testing, when the sample does not conform to the description provided, and when the test required is not fully specified, the laboratory makes every attempt to consult the client for further instruction before proceeding. The laboratory establishes whether the sample has received all necessary preparation, or whether sample preparation has yet to be performed. If the sample does not meet the sample receipt acceptance criteria listed in this standard, the laboratory:
 - Retains correspondence and/or records of conversations concerning the final disposition of rejected samples; or
 - 2) Fully documents any decision to commence with the analysis of samples not meeting acceptance criteria.
 - The condition of these samples is, at a minimum, noted on the chain of custody record or transmittal form, and laboratory receipt documents.
 - ii. The analysis data is/are appropriately "qualified" on the final report.
- d) The laboratory utilizes a permanent chronological record such as a logbook or electronic database to document receipt of all sample containers.
 - 1) This sample receipt log records the following:
 - Client/Project Name;
 - ii. Date and time of laboratory receipt;
 - iii. Unique laboratory ID code (see Calscience QSM Section 11.1); and
 - iv. Signature or initials of the person making the entries.
 - 2) During the login process, the following information is linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, that document becomes part of the laboratory's permanent records, easily retrievable upon request, and readily available to individuals who will process the sample. Note: The placement of the laboratory ID number on the sample container is not considered a permanent record.
 - i. The field ID code that identifies each container is linked to the laboratory ID code in the sample receipt log.
 - ii. The date and time of sample collection is linked to the sample container and to the date and time of receipt in the laboratory.
 - iii. The requested analyses (including applicable approved test method numbers) are linked to the laboratory ID code.
 - Any comments resulting from inspection for sample rejection are linked to the laboratory ID code.

- e) All documentation (i.e., memos or transmittal forms) that are conveyed to the laboratory by the sample submitter is retained.
- f) A complete chain of custody record form is maintained.

11.4 Storage Conditions

The laboratory has documented procedures and appropriate facilities to avoid deterioration, contamination, and damage to the sample during storage, handling, preparation, and testing; any relevant instructions provided with the item are followed. Where items must be stored or conditioned under specific environmental conditions, these conditions are maintained, monitored, and recorded.

- a) Samples are stored according to the conditions specified by preservation protocols:
 - Samples that require thermal preservation are stored under refrigeration at +/-2° of the specified preservation temperature unless method-specified criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C is acceptable.
 - 2) Samples are stored away from all standards, reagents, food, and other potentially contaminating sources. Samples are stored in such a manner to prevent cross contamination.
- b) Sample fractions, extracts, leachates, and other sample preparation products are stored according to Calscience QSM Section 11.4.a above or according to specifications in the test method.
- c) When a sample or portion of a sample needs to be held secure (for example, for reasons of record, safety or value, or to enable check calibrations or tests to be performed later), the laboratory has storage and security arrangements that protect the condition and integrity of the secured items or portions concerned.

11.5 Sample Disposal

The laboratory has standard operating procedures for the disposal of samples, digestates, leachates and extracts or other sample preparation products.

12.0 RECORDS

The laboratory maintains a record system to suit its particular circumstances and comply with any applicable regulations. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the test report for a minimum of five years.

There are two levels of sample handling: 1) sample tracking and 2) legal chain of custody protocols that are used for evidentiary or legal purposes. All essential requirements for sample tracking (e.g., chain of custody form) are outlined in Calscience QSM Sections 12.1, 12.2 and 12.3. Calscience has a written SOP that describes how it will carry out legal chain of custody for example, ASTM D 4840-95 and Manual for the Certification of Laboratories Analyzing Drinking Water, March 1997, Appendix A.

12.1 Record Keeping System and Design

The Calscience record keeping system allows historical reconstruction of all laboratory activities that produced the analytical data. The history of the sample is readily understood through the documentation. This includes inter-laboratory transfers of samples and/or extracts.

- a) The records include the identity of personnel involved in sampling, sample receipt, preparation, calibration or testing.
- b) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification, are documented.
- c) The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files.
- d) All changes to records are signed or initialed by responsible staff. The reason for the signature or initials is clearly indicated in the records such as "sampled by," "prepared by," or "reviewed by."
- e) All generated data, except those that are generated by automated data collection systems, are recorded directly, promptly, and legibly in permanent ink.
- f) Entries in records are not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors are made by one line marked through the error. The individual making the correction signs (or initials) and dates the correction. These criteria also apply to electronically maintained records.
- g) Refer to 10.6 for Computer and Electronic Data.

12.2 Records Management and Storage

- a) All records (including those pertaining to calibration and test equipment), certificates and reports are safely stored, and held secure and in confidence to the client. NELAP-related records are available to the accrediting authority.
- b) All records, including those specified in Calscience QSM Section 12.3, are retained for a minimum of five years from generation of the last entry in the records. The laboratory maintains all information necessary for the historical reconstruction of data. Records stored only on electronic media are supported by the hardware and software necessary for their retrieval.
- c) Records that are stored or generated by computers or personal computers have hard copy or write-protected backup copies.
- d) The laboratory has an established record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation storage and reporting.
- e) Access to archived information is documented with an access log. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.
- f) The laboratory has a plan to ensure that the records are maintained or transferred according to the clients' instructions (see 4.1.8.e of NELAC) in the event that a laboratory transfers ownership or goes out of business. In all cases, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

12.3 Laboratory Sample Tracking

12.3.1 Sample Handling

A record of all procedures to which a sample is subjected while in Calscience's possession is maintained. These include but are not limited to all records pertaining to:

- a) Sample preservation, including appropriateness of sample container and compliance with holding time requirement;
- b) Sample identification, receipt, acceptance or rejection, and log-in;
- Sample storage and tracking, including shipping receipts, sample transmittal forms (chain of custody form); and
- d) Documentation procedures for the receipt and retention of test items, including all provisions necessary to protect the integrity of samples.

12.3.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following is retained:

- All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- b) A written description or reference to the specific test method used, which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- c) Copies of final reports;
- d) Archived standard operating procedures;
- e) Correspondence relating to laboratory activities for a specific project;
- f) All corrective/preventive action reports, audits and audit responses;
- g) Proficiency test results and raw data; and,
- h) Results of data review, verification, and cross-checking procedures.

12.3.3 Analytical Records

The essential information associated with analyses, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- a) Laboratory sample ID code;
- b) Date of analysis and time of analysis if the method-specified holding time is 72 hours or less, or when time critical steps are included in the analysis, e.g., extractions, and incubations;
- c) Instrument identification and instrument operating conditions/parameters (or reference to such data):
- d) Analysis type;

- e) All manual calculations e.g., manual integrations;
- f) Analyst's or operator's initials/signature;
- Sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- h) Sample analysis;
- i) Standard and reagent origin, receipt, preparation, and use;
- j) Calibration criteria, frequency and acceptance criteria;
- k) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- Quality control protocols and assessment;
- m) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- n) Method performance criteria including expected quality control requirements.

12.3.4 Administrative Records

The following are maintained:

- a) Personnel qualifications, experience and training records;
- b) Records of demonstration of capability for each analyst; and
- A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

13.0 LABORATORY REPORT FORMAT AND CONTENTS

The results of each test, or series of tests carried out by the laboratory must be reported accurately, clearly, unambiguously and objectively. The results normally reported in a test report and include all the information necessary for the interpretation of the test results and all information required by the method used. Some regulatory reporting requirements or formats, such as monthly operating reports may not require all items listed below, however, Calscience will provide all the required information to their client for use in preparing such regulatory reports.

- a) Except as discussed in 13.b, each report to an outside client includes at least the following information (those prefaced with "where relevant" are not mandatory):
 - 1) A title, e.g., "Analytical Report," or "Test Certificate," "Certificate of Results" or "Laboratory Results";
 - Name and address of laboratory, and location where the test was carried out if different from the address of the laboratory and phone number with name of contact person for questions;

 Unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;

This requirement may be presented in several ways:

- The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or
- ii. Each page is identified with the unique report identification, the pages are identified as a number of the total report pages (example: 3 of 10, or 1 of 20).

Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report, and that the report contains a specified number of pages.

- 4) Name and address of client, where appropriate and project name if applicable;
- Description and unambiguous identification of the tested sample including the client identification code;
- Identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;
- 7) Date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours;
- Identification of the test method used, or unambiguous description of any nonstandard method used;
- 9) If the laboratory collected the sample, reference to sampling procedure;
- 10) Any deviations from (such as failed quality control), additions to or exclusions from the test method (such as environmental conditions), and any nonstandard conditions that may have affected the quality of results, and including the use and definitions of data qualifiers.
- 11) Measurements, examinations and derived results, supported by tables, graphs, sketches, and photographs as appropriate, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis; identify the reporting units such as µg/l or mg/kg;
- 12) When required, a statement of the estimated uncertainty of the test results;
- 13) A signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the certificate or report (however produced), and date of issue;
- 14) At the Calscience's discretion, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory;
- 15) At the Calscience's discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;
- 16) Clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc.; and

- 17) Clear identification of numerical results with values outside of quantitation limits.
- b) Where the certificate or report contains results of tests performed by subcontractors, these results are clearly identified by subcontractor name or applicable accreditation number.
- c) After issuance of the report, the laboratory report remains unchanged. Material amendments to a calibration certificate, test report or test certificate after issue may be made only in the form of a further document, or data transfer, including the statement "Supplement to Test Report or Test Certificate, serial number . . . [or as otherwise identified]", or equivalent form of wording. Such amendments meet all the relevant requirements of the NELAC Standard.
- d) Calscience notifies clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any calibration certificate, test report or test certificate or amendment to a report or certificate.
- e) The laboratory will, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, follow documented procedures that ensure that the requirements of this Standard are met and that confidentiality is preserved.
- f) Calscience will certify that all its NELAC-certified test results reported meet all requirements of NELAC or provide reasons and/or justification if they do not.

14.0 SUBCONTRACTING ANALYTICAL SAMPLES

When Calscience subcontracts work whether because of unforeseen circumstances (e.g. workload, need for further expertise or temporary incapacity) or on a continuing basis (e.g. through client direction, contractual arrangement or permanent subcontracting), this work shall be placed with a laboratory accredited under NELAP for the tests to be performed or with a laboratory that meets applicable statutory and requirements for performing the tests and submitting the results of tests performed. The laboratory performing the subcontracted work shall be indicated in the final report.

- a) Calscience will advise its client via written, facsimile or e-mail notification of its intention to subcontract any portion of the testing to another party in cases when unforeseen circumstances occur. Calscience shall gain approval by the client preferably in writing, facsimile or via e-mail response.
- b) Calscience may subcontract samples on a continuing basis without written, facsimile or e-mail notification under the following (but not limited to) cases:
 - 1) Client direction or instruction
 - 2) Contractual specification or requirement
 - 3) Project historical precedent
- c) Calscience retains records demonstrating that the above requirements have been met.

15.0 OUTSIDE SUPPORT SERVICES AND SUPPLIES

Calscience does not procure outside services and supplies, other than those referred to in this Manual.

16.0 INQUIRIES AND COMPLAINTS

Calscience SOP-T018 addresses the policies and procedures for the resolution of inquiries and complaints received from clients or other parties about the laboratory's activities. Where an inquiry or complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this manual or otherwise concerning the quality of the laboratory's calibrations or tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with NELAC Section 5.3.1. Records of the complaint and subsequent actions are maintained.

NELAC APPENDICES

APPENDIX A - REFERENCES

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EPA/600/4-90/031 Manual for Evaluation of Laboratories Performing Aquatic Toxicity Tests, Office of Research and Development, Washington, DC, 1991.

EPA/600/3-88/029 Protocol for Short-term Toxicity Screening of Hazardous Wastes, Office of Research and Development, Washington, DC, 1991.

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"Glossary of Quality Assurance Terms and Acronyms," Quality Assurance Division, Office of Research and Development, USEPA.

"Guidance on the Evaluation of Safe Drinking Water Act Compliance Monitoring Results from Performance Based Methods," September 30, 1994, Second draft.

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ISO Guide 3534-1: "Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms."

ISO Guide 7218: Microbiology - General Guidance for Microbiological Examinations.

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ISO Guide 9000: 1994. Quality management and quality assurance standards - Guidelines for selection and use.

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ISO Guide 9002: 1994. Quality systems - Model for quality assurance in production and installation.

ISO/IEC Guide 2: 1986. General terms and their definitions concerning standardization and related activities.

ISO/IEC 17025: 1999. General requirements for the competence of calibration and testing laboratories.

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Manual for the Certification of Laboratories Analyzing Drinking Water, Revision 4, EPA 815-B-97-001.

Manual of Method for General Bacteriology, Philipp Gerhard et al., American Society for Microbiology, Washington, D.C. 1981.

Performance Based Measurement System, EPA EMMC Method Panel, PBMS Workgroup, 1996.

APPENDIX B - GLOSSARY

The following definitions are used in the text of Quality Systems. In writing this document, the following hierarchy of definition references were used: ISO 8402, ANSI/ASQC E-4, EPA's Quality Assurance Division Glossary of Terms, and finally definitions developed by NELAC. The source of each definition, unless otherwise identified, is the Quality Systems Committee.

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority: The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation. (NELAC) [1.5.2.3]

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Analysis Duplicate: The second measurement of the target analyte(s) performed on a single sample or sample preparation.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Analytical Reagent (AR) Grade: Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society. (Quality Systems)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Audit: A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch: Environmental samples, which are prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample: A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method: A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard: A substance or reference material used to calibrate an instrument. (QAMS)

Certified Reference Material (CRM): A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody Form: A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions compromised samples are not analyzed. If emergency situations require analysis, the results must be appropriately qualified. (NELAC)

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors: or
- Additional cleanup procedures. (NELAC)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)

Desorption Efficiency: The mass of target analyte recovered from sampling media, usually a sorbent tube, divided by the mass of target analyte spiked on to the sampling media expressed as a percentage. Sample target analyte masses are usually adjusted for the desorption efficiency. (NELAC)

Detection Limit: The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA- QAD)

Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR Part 136)

Inspection: An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ ASQC E4-1994)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (NELAC)

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Laboratory: A body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC)

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. See also Method Detection Limit, Detection Limit, and Quantitation Limit. (Analytical Chemistry, 55, p. 2217, December 1983, modified)

Manager (however named): The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

- Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
- Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous Liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material.
 Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.
- Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
- Air: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the
 extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube,
 impinger solution, filter or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)

May: Denotes permitted action, but not required action. (NELAC)

Media: Material that supports the growth of a microbiological culture.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B)

Must: Denotes a requirement that must be met. (Random House College Dictionary)

National Accreditation Database: The publicly accessible database listing the accreditation status of all laboratories participating in NELAP. (NELAC)

National Environmental Laboratory Accreditation Conference (NELAC): A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAC.

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Objective Evidence: Any documented statement of fact, other information, or record, either quantitative or qualitative, pertaining to the quality of an item or activity, based on observations, measures, or tests that can be verified. (ASQC)

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS): A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, and analysis) which must be strictly followed. (EPA- QAD)

Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method. (NELAC)

Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample: An uncontaminated sample matrix with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ ASQC E-41994)

Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence. (NELAC)

Range: The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30- 2.1)

Reference Method: A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Reference Toxicant: The toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results (see Chapter 5, Appendix D, Section 2.1.f). (NELAC)

Replicate Analyses: The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Requirement: Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Sampling Media: Material used to collect and concentrate the target analytes(s) during air sampling such as solid sorbents, filters, or impinger solutions.

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM): A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named): The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director: Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Testing Laboratory: Laboratory that performs tests. (ISO/ IEC Guide 2 - 12.4)

Test Sensitivity/Power: The minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis (see Chapter 5, Appendix D, Section 2.4.a). (NELAC)

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Validation: The process of substantiating specified performance criteria. (EPA- QAD)

Verification: Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: A well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Sources:

American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996

American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991

ANSI/ASQC E4, 1994

ANSI N42.23- 1995, Measurement and Associated Instrument Quality Assurance for Radiobioassay Laboratories

International Standards Organization (ISO) Guides 2, 30, 8402

International Vocabulary of Basic and General Terms in Metrology (VIM): 1984. Issued by BIPM, IEC, ISO and OIML

National Institute of Standards and Technology (NIST)

National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards

Random House College Dictionary

U.S. EPA Quality Assurance Management Section (QAMS), Glossary of Terms of Quality Assurance Terms, 8/31/92 and 12/6/95

U.S. EPA Quality Assurance Division (QAD)

40 CFR, Part 136

Webster's New World Dictionary of the American Language

APPENDIX C - DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR DEMONSTRATION OF CAPABILITY

A demonstration of capability (DOC) must be made prior to using any test method, and at any time there is a change in instrument type, personnel or test method. (See NELAC 10.2.1.)

Note: Where tests are performed by specialized "work cells" (a well-defined group of analysts that together perform the method analysis), the work cell as a unit meets the above criteria and this demonstration is fully documented.

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids and air. However, before any results are reported using this method, actual sample spike results may be used to meet this standard, i.e., at least four consecutive matrix spikes within the last twelve months. In addition, for analytes that do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations shall be documented through the use of the form in this appendix.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, are performed if required by mandatory test method or regulation. Note: For analytes for which spiking is not an option and for which quality control samples are not readily available, the 40 CFR approach is one way to perform this demonstration. The laboratory documents that other approaches to DOC are adequate, and this is documented in the laboratory's Quality Manual.

- a) A quality control sample is obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.
- b) The analyte(s) is diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.
- c) At least four aliquots are prepared and analyzed according to the test method either concurrently or over a period of days.
- d) Using all of the results, the mean recovery (\overline{X}) is calculated in the appropriate reporting units (such as $\mu g/L$) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory will assess performance against established and documented criteria.
- e) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are no established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.
- f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.

- 1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.
- 2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

C.2 CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee (see Calscience QSM Section 6.3 and 12.3.4.b.).

Demonstration of Capability Certification Statement

Date: Laboratory Name: Laboratory Address: Analyst(s) Name(s):		Pageof
Matrix: pure water, soil, air,	solid, biological tissue)	
Method number, SOP#, Rev #, and Analy (examples: barium		Measured Parameters: 6010, benzene by 8021, etc.)
We, the undersigned, CERTIFY that:		
1. The analysts identified above, using the analyses of samples under the National Edemonstration of Capability.		
2. The test method(s) was performed by the	ne analyst(s) identified on th	nis certification.
3. A copy of the test method(s) and the lab	poratory-specific SOPs are	available for all personnel on-site.
4. The data associated with the demonstrate explanatory (1).	ation capability are true, acc	curate, complete and self-
5. All raw data (including a copy of this cer analyses have been retained at the facility available for review by authorized assessor	, and that the associated in	
Technical Director's Name and Title	Signature	Date
Quality Assurance Officer's Name	Signature	Date
This certification form must be completed	each time a demonstration	of capability study is completed.
True: Consistent with supporting data. Accurate: Based on good laboratory practices of Complete: Includes the results of all supporting Self-explanatory: Data properly labeled and sto	performance testing.	

(Note: Form may be modified so long as the essential items are included in the updated form)

(1)

APPENDIX D - ESSENTIAL QUALITY CONTROL REQUIREMENTS

The quality control protocols specified by the laboratory's method manual (10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals.

All quality control measures shall be assessed and evaluated on an ongoing basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter 5, e.g., Section 5.4, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing.

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

a) Negative Controls

- Method Blanks Shall be performed at a frequency of one per preparation batch of samples per matrix type. The results of this analysis shall be one of the QC measures to be used to assess the batch. The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if
 - i) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or
 - ii) the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

b) Positive Controls

- 1) Laboratory Control Sample (LCS) (QC Check Samples) Shall be analyzed at a minimum of 1 per preparation batch of 20 or less samples per matrix type, except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to assess the batch. NOTE: The matrix spike (see 2 below) may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS.
- 2) Matrix Spikes (MS) Shall be performed at a frequency of one out of every 20 samples per matrix type prepared over time, except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.
- 3) Surrogates Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate

is not available. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

4) If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene, and PCBs in Method 608), the test method has an extremely long list of components or components that are incompatible, a representative number (minimum of 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit-specified analytes, and other client-requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

D.1.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates - Shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document its procedure to select the use of appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

D.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

- a) Demonstration of Analytical Capability (Section 10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.
- b) Calibration Calibration protocols specified in Section 9.4 shall be followed.
- c) Proficiency Test Samples The results of such analyses (4.2.j or 5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.1.4 Detection Limits

The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the mandated test method or applicable regulation, e.g., Method Detection Limit (MDL). If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

- a) A detection limit study is not required for any component for which spiking solutions or quality control samples are not available such as temperature.
- b) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).
- c) Detection limits must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

- All samples processing steps of the analytical method shall be included in the determination of the detection limit.
- e) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.
- f) The laboratory must have established procedures to relate detection limits with quantitation limits.
- g) The test method's quantitation limits must be established and must be above the detection limits.

D.1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.1.6 Quality of Standards and Reagents

- a) The source of standards shall comply with 9.2.
- b) Reagent Quality, Water Quality and Checks:
 - 1) Reagents In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.
 - 2) Water The quality of water sources shall be monitored and documented and shall meet method specified requirements.
 - 3) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.

D.1.7 Selectivity

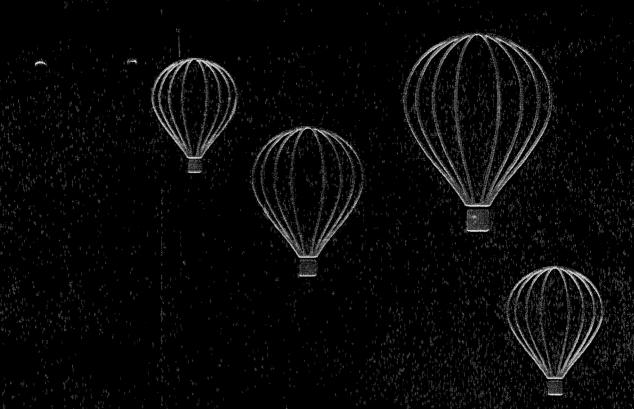
- a) Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory shall develop and document acceptance criteria for retention time windows.
- b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.
- c) The laboratory shall document acceptance criteria for mass spectral tuning.

D.1.8 Constant and Consistent Test Conditions

- a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.
- b) Glassware Cleaning Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

END OF DOCUMENT



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"Quality....the totality of features and characteristics of a product or service that bear on its ability to satisfy stated or implied needs."

"Quality Assurance....all those planned and systematic actions necessary to provide adequate confidence that a product or service will satisfy given requirements for quality."

Excerpts from ISO 8402

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APPENDICES

- A. Definitions & Terms
- B. List of Standard Operating Procedures (SOPs)

REFERENCES



This Quality Manual was designed to meet 2003 NELAP (National Environmental Laboratory Accreditation Program) standards and supports assessment programs and/or certifications with the

following agencies:

Certifying Agency	ATL Certificate #	Basis of Certification/Approval
California DOH (Primary NELAP)	02110CA	Onsite assessment (biennial) and WP PTs
Florida DOH (Primary NELAP)	E87680	Onsite assessment (biennial) and SOP Review
New Jersey DEP (Primary NELAP)	CA004	Onsite assessment (biennial) and SOP Review
Louisiana DEQ	02089	SOP Review, WP PTs, Secondary NELAP
New York State DOH	11291	NY PTs, QAM (Secondary NELAP)
State of Utah DOH	9166389892	QAM, Secondary NELAP
U.S. Navy NFESC/IR/QA	NA	DOD Quality System Manual for Environmental Laboratories v.2/ Recognition of NELAP Accreditation
Arkansas DEQ	03-084-0	QAM, PT, MDL Review
Pennsylvania State Dept. Health	68-690	

MANAGEMENT QUALITY POLICY STATEMENT

At ATL, we strive to be the **BEST** in everything that we do. Our very existence is based on our continued ability to provide innovative, dependable, and cost effective environmental services to our clients. We **CARE** about our clients as well as our co-workers and manage our daily activities to build relationships based on mutual **TRUST**, **HONESTY**, and **RESPECT**. We are **LEADERS** in our field and accept the risks associated with building new frontiers in our professional lives. Our strength comes from our **TEAMS** for through them we can achieve our goals. This Quality Assurance Manual defines and documents the core systems surrounding good professional as well as laboratory best practices for all staff. The management signatures below represent our commitment to continually define, assess, and improve the quality systems, which provide the basic infrastructure in support of these goals.

Linda L. Freeman Smile Tuman	7/10/06
CEO, Laboratory Director (Technical Director I)	Date
Heidi C. Hayes Mula Mano	7/10/06
VP, Director of Business Development (Technical Director II)	Date
Melanie A. Levesque Wong (alaball	7/10/06
Quality Assurance Manager	Date

The Air Toxics Limited Quality Assurance Manual is effective as of the date of the signature of the Laboratory Director.

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1.0 INTRODUCTION

The Air Toxics Limited (ATL) Quality Manual describes the Quality Assurance (QA) program and Quality Control (QC) procedures used to ensure that data of known and documented quality are produced. It is designed to be used as a manual that outlines the process by which we ensure that the customer expectations are met, and hence, the quality goal is met. ISO/IEC Guide 17025-General Requirements Competence of Calibration and Laboratories are incorporated wherever possible, however the primary guidance document is Chapter 5: Quality Systems as published in the June 5, 2003 NELAP Standard.

The Quality Manual contains a discussion of the following topics:

Introduction: The quality objective is discussed along with management and information systems in support of the objective.

Organization: Staff qualifications and responsibilities, management organization, laboratory facilities, and equipment are detailed in this section.

Quality Assurance Program: This section deals with project management, standard operating procedures, staff members' training, evaluation and documentation of adherence to quality assurance and quality control requirements, corrective action system, and health and safety.

Quality Objectives: This section explains the quality control parameters and procedures, procedures to establish limits of detection and quantitation and perform calibrations, traceability, and preparation of standards.

Sample Handling: Sampling containers, preservation and Chain-of-Custody requirements, sample receiving and tracking procedures, internal custody, storage and disposal are discussed.

Analytical Methods and Procedures: In this section, a brief method description is given for all analytical procedures carried out at ATL. The limit of quantitation concentrations, quality control acceptance criteria and method modifications are provided as well.

Data Review and Reporting: This section explains the procedures involved in data collection/reduction, data review, and final report production. Hardcopy and electronic data production, data flagging, and data storage are also discussed in this section.

Establishing Acceptance Criteria: The control chart program is outlined in this section along with generating and evaluating in-house statistical limits.

Preventative Maintenance: Routine maintenance, service contracts, and control of miscellaneous monitoring equipment are explained briefly.

Assessments and PT Samples: A brief explanation of internal and external assessments programs and NELAP PT samples program is provided.

Computer and Software Systems: This section of the quality manual deals with the management of computer and software systems. Data storage, back-up routines, and internal software validation efforts are included.

Control of Purchased Items: Control of purchased items and external services as well as the purchase requisition system are outlined in this section.

Project Management System: This part of the quality manual gives a brief description of steps to ensure that the customer expectations are met once the project is undertaken.

1.1 QUALITY OBJECTIVE

Air Toxics Limited is committed to producing data that meet or exceed the client's measurement needs. Customer satisfaction is the motivating force behind most of the ATL processes. An underlying network of systems designed to define, document, and process each individual customer's need supports this primary objective. This systems network includes Marketing/Sales, Project Management, IT, Laboratory Production, Support Services, Technical Services, Quality Assurance, and Finance. Each of these operational areas is organized around an empowered work team accountable for delivering an automated, on time and defensible result.

We believe the ultimate responsibility for quality resides at the team level. Every team member has the responsibility and authority to suspend a process if it appears that the quality objectives are not being met. Analytical team members are the quality objectives informed of documented Standard Operation Procedures (SOPs) and project related information systems (ATL's Project Profiles and Project Requirement tables). Team members work closely with the Project Management and QA Departments to ensure that the quality objectives are met.

1.2 QUALITY MANAGEMENT SYSTEM

The role of the ATL management team is to help ensure that the quality objective is met through a continuous and reiterative program of process improvements. The management team consists of Business Directors, Department Managers and Team/Task Leaders.

The primary role of the management team is to establish performance goals at the corporate and team levels as well as to develop tools capable of producing quantifiable measures of performance against these goals (e.g., customer satisfaction index, sales quotas, report turn around time, net profit, days to complete corrective actions, etc.). A secondary role of management is to help ensure that the work environment and facilities promote

continued development of empowered work teams through facilities management and programs for recruiting, training, and retaining qualified staff.

Quality Assurance Management: The role of the ATL Quality Assurance team is to help ensure that the systems described above are designed, documented, and operating in accordance with the quality objectives. This is accomplished via coordination and dissemination of internal and external assessment information, review of SOPs to document variances taken to published methods, monitoring of the Quality Manual to consistency with actual practices, maintenance of an ongoing Corrective Action Program with quarterly reports to management. and a leadership role in employee training programs. A secondary function of the QA team deals with data review and other quality control related programs.

The QA team is free from any commercial, financial, or production pressures when making assessments or decisions regarding the quality of work produced or effectiveness of the quality systems. The Quality Assurance Manager reports directly to the President in order to maintain independence from business operating units and facilitate communications regarding quality related issues.

Communication between the QA team and other management teams occurs on a regular basis via weekly status meetings. Information regarding outstanding corrective action items, upcoming assessments, assessment results or general observations are brought up and documented via a database of agenda notes. This database along with the corrective action database and ATLAS database compiles a 'Quarterly Quality Assurance Status Report', which is distributed to the entire management team for review.

Sales and Project Management: The role of the ATL Sales and Client Services teams is to effectively document and communicate the needs of the customer. These teams represent the

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customer in all matters and serve as a liaison between the customer and the Technical Services. Laboratory, Support Services, Finance, and Quality Assurance areas. The ATL Marketing and Client Services teams ensure that client needs are matched by laboratory resources. communication linkages exist between the lab Department Managers, Team Leaders and the ATL Marketing and Client Services teams. Information regarding customer needs flows into all ATL systems via these two teams. Interactions may be as complex as Quality Assurance Project Plan (QAPP), contract or Scope of Work (SOW) review or as simple as processing shipments of canisters and other sampling media. Project specifics are documented and stored via an interactive database that assigns a unique identifier for every reference.

Sample Receiving: The goal of the department is to enable every sample to be received and processed into a unique laboratory Work Order within 24 hours of sample receipt. Sample non-conformities are communicated to the clients in the same time frame. Custody information relating to sample receipt, a copy of the sample receipt summary, and an example report format is emailed or faxed to the client for review and comments.

Laboratory Management: Laboratory management is divided into work teams equipped with necessary resources to complete the sample analysis, review, data reporting and creation of all electronic data packages, which include email EDD and eCVP on CD-ROM. The laboratory work teams are responsible for verifying the quality of electronic deliverables by reviewing a percentage of the product. In this way, team members are easily able to accept the control and accountability for quality. The Support Services team is responsible for cleaning, assembling, certification and shipping all sampling media. The primary responsibility of the Team or Task

The primary responsibility of the Team or Task Leaders is to monitor customer needs versus resource availability. Staff and equipment management are carefully balanced with customer needs. The goal for each team is to deliver defensible data within the time frame promised to the client. The Team or Task Leaders review daily sample receipt work lists to determine that the laboratory has adequate resources to perform the work. In those cases where either the technical or sample capacity demands cannot be met, the Team or Task Leader works with the Client Services Representative and the client to provide a solution via inside resource re-allocation or outside subcontracting. The ATLAS laboratory automation system creates and tracks special analytical lists, deliverables, or Turn Around Time (TAT) requests which are automated via customized linkages (work tools) into the centralized Structured Query Language (SQL) database. Performance measurements against the goal are routinely monitored using the same SQL Performance and quality related database. information is shared with team members during Project or client related team meetings. information resides both in the project management module and in sample tracking modules, reducing the need for relying on verbal communication of project specifics to the team. The Team and Task Leaders report to a Department Manager who reports to the President.

The remaining team positions are divided into three levels:

1) Senior Scientist (Lab Personnel) or Senior Associate (non-Lab Personnel)

This is the highest level professional position reporting to a Director, Manager, or Team Leader. The Senior Scientist or Senior Associate works independently at a company wide level. In addition to all of the responsibilities of the Scientist described below, the Senior Scientist or Senior Associate is recognized within the company as an expert in his/her field. He or she is often asked to work outside of the team whenever the need arises, and is able to demonstrate above average leadership skills. Senior Scientists or Senior Associates are

responsible for method development activities and take a lead role in proprietary software and hardware design and testing. High profile projects or client relations, including more intricate analyses and data interpretations, are assigned to a Senior Scientist to oversee. The Senior Scientist or Senior Associate maintains knowledge at the level of Masters Degree or equivalent with a minimum of 5 years of analytical environmental experience.

Scientist (Lab Personnel) or Associate (Non-Lab Personnel)

The Scientist or Associate works independently at the team level. A Scientist or Associate demonstrates a high level of skill, judgement, problem solving ability, and is able to independently perform troubleshooting. The responsibilities of a Scientist/Associate include: scheduling of work, providing routine as well as non-routine bench level activities in a highly efficient manner, writing SOPs, reviewing data, performing non-routine instrument maintenance and troubleshooting, and representing the team during internal/external assessments. Individuals in this position play a lead role in monitoring health and safety on the team, and acting as a resource or trainer. A Scientist or Associate must have a Bachelor's Degree and a minimum of analytical three years of environmental experience.

Analyst (Lab Personnel and Non-Lab Personnel)

The Analyst works under the direct supervision of a Scientist, Senior Scientist, Team Leader and/or Manager at all times. He/she follows a specific formal training program to learn the necessary skills required of the position and demonstrates the ability to recognize problems and to seek assistance. The primary responsibility of an Analyst is to follow written laboratory SOPs in an efficient and wellinformed manner. The Analyst performs routine maintenance the equipment, on standards, and performs all relevant bench level activities. The minimum qualification for a laboratory analyst is a Bachelor's Degree.

Each team has a mix of Scientists and Analysts. Each Team Leader coordinates the activities of the respective team, serves as a resource to the Analysts and Scientists, communicates the corporate objectives to the team, and monitors team progress against the quality objective, which is customer satisfaction. A Scientist may be assigned as 'team lead' in a subset of team As 'team lead' the Scientist is activities. responsible for all the technical activities of the assigned area, oversees both the quality and quantity of work produced, and serves as resource for the Analysts. The Analyst performs all of the routine activities and quality checks (i.e., makes sure the customer expectations are met).

Every team member is empowered to make sure that the customer expectations are met, is trained in the elements of the quality process, and has the responsibility and authority to stop or suspend a process when the quality objective is in jeopardy.

Information Technology:

The Information Technology (IT) team is responsible for the design and maintenance of the SQL server based data system. Its primary goal is to ensure that customer satisfaction is achieved by the way information is transferred, processed, or queried. This includes systems relating to telephone service, e-mail service, Internet access, project management, data acquisition, assessment trails, data security, and automated data reporting linkages. The group consists of the IT Manager, and one full time programmer. Around-the-clock, system support is achieved via a combination of in-house and contract support. Additional programmers are hired on a project specific basis. All of the ATL information systems are designed, coded, and tested in house and as such, are proprietary in nature. The IT Manager reports to the President.

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Financial Management: The quality systems rely on bottom line profitability to provide strength to the framework that produces quality results. The ATL Finance team is responsible for monitoring the profitability of all operations. Customer satisfaction goals are built into budgeting, purchasing, invoicing, employee compensation and benefits programs, collections, contracts, insurance, and banking. The primary goal of the team is help ensure bottom line achieving the profitability while objective, which is customer satisfaction. The group consists of a Controller, a Finance Associate, a Credit & Collection Associate and an Accounts Payable/Purchasing Associate. Finance Team Leader reports to the President.

Data Integrity **Procedures:** Since commitment to data integrity is a vital component for credibility of our core product, Air Toxics Limited cannot function as a business entity without a clear definition of ethical expectations for all employees. Integrity is defined as the ability to discern right from wrong, and the commitment to do what is right, good and proper. Data integrity procedures relating to generation of analytical reports are built into the systems via the operational SOPs, which describe appropriate practices. Additional systems and training programs that safeguard strict adherence to the SOPs ultimately ensure that data integrity procedures are employed. Intentional fraud will be grounds for severe reprimand and/or termination of complicit employees. In addition, employees who witness or are otherwise aware of data integrity violations, even if they are not a party to such acts, are expected to immediately report these lapses to their Team/Task Leader, their Department Manager or to a member of the Board of Directors.

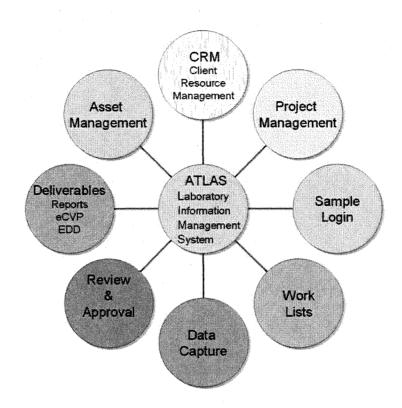
Data integrity training is conducted within a variety of frameworks and is mandatory for all Air Toxics Limited employees. New employees read both the LQAP and the Employee Handbook to properly orient ethical expectations. In addition, within one month of date of hire there is basic training provided by the Quality Assurance

Department to familiarize new employees with principles of documentation to pre-empt practices that would call into question the data integrity procedures of the laboratory. The Chief Executive Officer of Air Toxics conducts a monthly Standards of Conduct presentation that defines data integrity expectations, potential penalties and consequences for lapses of integrity for new employees. In addition, the CEO conducts a yearly ongoing Ethics Training II for remainder of the employees. Inappropriate Lab Practices Class (also done on a yearly basis) defines allowable parameters for the lab to assure defensibility and to define illegal The purpose of all training is to provide specific examples of data integrity expectations that are relevant to actual job functions. Employees document the training in their training records (see Section 3.2.1).

1.3 INFORMATION MANAGEMENT SYSTEM

Information is stored in the Air Toxics Laboratory Automation System (ATLAS) databases using ATL designed hardware and proprietary software. This in-house Laboratory Information Management System (LIMS) is an evolving development project designed to find more efficient means to meet the customer needs. Each client contact (telephone call, quote, shipping request, or inquiry) is stored in a database, which can be queried for sample log-in, project backlogs, project TAT or revenue statistics.

Some modules are designed to track nontraditional information such as the sample history of individual canisters, number of reports completed per analyst per shift, and overdue work by reason code. These types of information directly affect the ability of the management team to provide quality process improvements. Some non-traditional calculations such as the boiling point distribution of a hydrocarbon background, **EPA** difference rounding, and percent calculations have been made available at the bench. This type of information directly affects the ability of the individual employee to meet the quality objectives.





2.0 ORGANIZATION

2.1 STAFF QUALIFICATIONS AND RESPONSIBILITIES

ATL's management organization includes the Board of Directors comprising four core areas: Operations, Finance, IT, and Sales. In addition there are Department Managers. Managers and Team/Task Leaders. Each operating area is either lead by a Department Manager or a Team/Task Leader. Due to the size and complexity of the main laboratory, Department Managers and Team or Task Leaders are required. Most Managers and Team/Task Leaders report to a member of the Board of Directors. One of the Directors is designated as the Vice President. If the President is absent, the Vice President may fulfill the responsibilities as President. addition, if the primary Technical Director is absent the second Technical Director will fulfill the responsibilities. If the OA Manager is absent, the Technical Directors may fulfill OA responsibilities. In the absence of a Manager or Team/Task Leader, one of the Directors will name an interim successor.

LINDA L. FREEMAN CHIEF EXECUTIVE OFFICER AND LABORATORY (TECHNICAL) DIRECTOR (1)

Ms. Linda L. Freeman is the Technical Director and the Chief Executive Officer of ATL providing leadership that ensures the founding mission and core values of the company are put into practice. Ms. Freeman leads programs relating to the development of long range strategy, quality systems, and financial infrastructure. As Technical Director responsibilities her include: (1). the administrative review of laboratory operations and qualifications for the technical positions, ensuring and documenting initial and ongoing proficiency, and oversight of the Quality systems. She holds a Bachelor's Degree from Boston College and a Master's Degree in Chemistry from the University of WisconsinMadison. Ms. Freeman has over 20 years of combined environmental experience and 17 years of laboratory business management experience.

BRAD MOSAKOWSKI PRESIDENT

Mr. Mosakowski is the President of ATL and represents the partnership in all matters. Mr. Mosakowski provides day-to-day leadership and management of programs for overseeing the processes and resources necessary for establishing long-range service objectives, plans and policies, in cooperation with the CEO and Board of Managers. responsible for the measurement and effectiveness of both internal and external processes by providing accurate and timely feedback on the operating condition of the company. In addition, Mr. Mosakowski also directs the definition and operation of the laboratory production by fostering a successoriented and accountable environment within the company. A critical component of which is his ability to motivate and lead a high performance management team capable of meeting both customer service and bottom line financial objectives. Mr. Mosakowski has over years of combined environmental 15 laboratory experience.

HEIDI C. HAYES VICE PRESIDENT, DIRECTOR OF BUSINESS DEVELOPMENT AND TECHNICAL DIRECTOR (2)

Ms. Heidi C. Hayes is the Vice President, the Director of Business Development and Technical Director (2) of ATL. Ms. Hayes is responsible for developing sustainable customer relations by providing customized solutions through technical leadership in marketing, sales and service. She is the key technical interface between laboratory services and major clients. Ms. Hayes plans, develops, and establishes policies and objectives for developing a more technical marketing, sales and service organization by performing the

following duties personally or through subordinate managers; provides the technical leadership, management and vision necessary to ensure the company has the proper operational controls, administrative procedures and human resource management in place to meet customer need and quality objectives. Ms. Hayes holds a Bachelor's Degree in Chemistry and Mathematics from Luther College and a Master's Degree in Chemistry from the Colorado School of Mines.

MELANIE LEVESQUE OUALITY ASSURANCE MANAGER

Levesque Ms. Melanie develops and supervises programs intended to ensure that the laboratory is producing data of known and acceptable quality. Ms. Levesque oversees QC activities including various independent checks of laboratory systems, SOP generation, and corrective action procedures, as well as monitoring laboratory certification programs. Ms. Levesque has documented training in the approved methods and can verify that the laboratory is following SOPs. Ms. Levesque maintains independence from the operations by not engaging in production activities and reports directly to the Vice President. The OA Department conducts a vearly independent audit of the quality systems and methods criteria, and notifies laboratory directors of deficiencies via a written quarterly status Ms. Levesque holds a Bachelor's degree in Chemistry and a Master of Science degree in Analytical Chemistry both from Rochester Institute of Technology, followed by six years of environmental laboratory experience. Ms. Levesque has worked in a variety of positions including 2 years as a HPLC chemist, 2.5 years as a GC/MS chemist, and has 4.5 years of laboratory supervisory experience.

NATHAN SHAFER LABORATORY DEPARTMENT HEAD

Mr. Nathan Shafer is the Department Manager for the Volatile Organic Compound (VOC) GC/MS analysis group. This department is responsible for all analyses via methods TO-14A/15, VOST methods 0030 and 0031, TO-17, and all VOC pptv work in the area of vapor intrusion. Mr. Shafer is responsible for managing and overseeing all processes and resources involved in the daily operations of the VOC department. In addition, he provides technical support to client services, sales, and the department; he is also responsible for coaching and training team members, data review, scheduling, and conferencing. Mr. Shafer has been employed by Air Toxics since 1997. His experience comes from roles such as GC/MS chemist, team leader, and project development chemist. Mr. Shafer holds a dual degree from Claremont McKenna College in the fields of chemistry and psychology.

SEPIDEH SAEED LABORATORY DEPARTMENT HEAD

Ms. Sepideh Saeed is the Department Manager for the GC, HPLC and GC/MS semi volatiles analysis, which includes EPA Method TO-3/TO-12, ASTM D-1945/1946, 25C/3C, TO-14A Direct Inject, Extractions, Headspace, ASTM D-5504, TO-13A. 5/CARB430, TO-11, Method 0011, PM10, TSP, NIOSH, Siloxanes, Pesticide and PCB Analytical Group. She is responsible for managing and overseeing all processes resources involved in the daily operations of SVOC department. In addition, she serves as Team Leader for the Reporting Team. She provides technical support to client services, sales, and the department and is also responsible for supporting both the Senior Scientist and the Task Leader in managing staff, production and technical matters. Ms. Saeed has 14 years of laboratory experience as a GC, HPLC, GC/MS and extraction chemist and 4 years of supervisory experience and has been employed at Air Toxics since 1998. Ms. Saeed has a B.S. Degree in Biochemistry from University of California, Davis.

JEFFREY TECSON SUPPORT SERVICES TEAM LEADER

Mr. Jeffrey Tecson is the Team Leader for the Support Services Team. This team is responsible for certifying and cleaning of Summa, Silco and Silonite Canisters. Other responsibilities include preparation of TO-17 tubes, VOST/SMVOC tubes for Methods 0030 and 0031. Mr. Tecson has 4 years experience in doing bench work for Support Services; currently Mr. Tecson is spending 25% of his time on the bench. Mr. Tecson has an A.S. Degree in Computer Technology Heald College in Rancho Cordova, CA; he also has 10 years management experience.

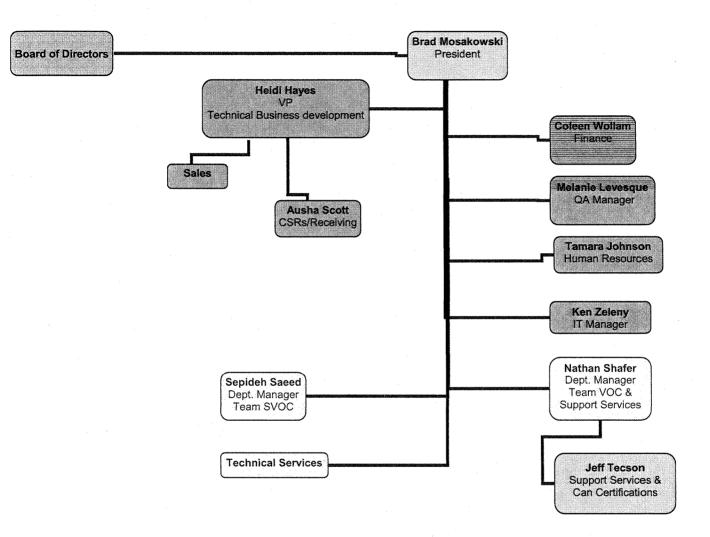
AUSHA SCOTT CLIENT SERVICES/RECEIVING TEAM LEADER

Ms. Ausha Scott is the Team Leader for the Client Services and Login/Receiving responsible Departments. She is overseeing the project management functions, including client relations and technical support. In addition, she directs the daily activities of the Login/Receiving team. Ms. Scott has 6 years of environmental laboratory experience in a variety of positions including GC/MS chemist and client service representative. Ms. Scott holds a Bachelor's degree in Marine Biology from University of California, Santa Cruz.

INFORMATION TECHNOLOGY MANAGER

Mr. Zeleny is the Information Technology Manager for the Group. IT responsibilities include database management, software development and Mr. Zeleny has network management. over 17 years experience with computer and technology functions in both large and small organizations. His experience also includes 5 years as a Sr. Systems Architect and then as a Manager of the Development Prior to this, Mr. Zeleny has worked as a Sr. NT Systems Engineer, IT Supervisor, Network Administrator and Sr. Technical Support Analyst. Zeleny has been employed at Air Toxics Limited since August, 2005.

Exhibit 2.1. ATL Management Organization



2.2 FACILITIES

The ATL laboratory occupies 30,000 square feet of space in Folsom, California with approximately 6,000 square feet of office space. The single story building is custom designed to suit the specifications of an air laboratory. Design criteria included floor segregation plans to accommodate conflicting tests and provide an environment that is conducive for cross-functional work teams. The main instrumentation laboratory is based on an "open" concept in which walls are removed to promote a sense of community and teamwork. Wide hallways with alcoves are designed to encourage congregation and discussion. The number of private offices is minimized that barriers SO management and staff are removed. Elements of the quality system are evident throughout the facility design.

Sample receiving occupies approximately 950 square feet. There is sufficient floor space to receive, unpack, and tag up to 150 SummaTM canisters per day. The main laboratory is centrally located and houses twenty GC/MS systems, eight GCs, and a network of computers.

A caged canister storage area was constructed on one side of the laboratory to securely hold all canister and Tedlar bag samples. An isolated negative pressure room was designed for solvent handling and extraction activities. Approximately 1000 square feet of airconditioned space is designated for research and development activities, and a work shop/tooling area. Sorbent tube preparation and canister cleaning operations are located in segregated areas. Long-term file storage occurs off site. A local document storage and retrieval service picks up files for storage.

Files are kept in bar coded boxes making retrieval easier. Typically a file can be retrieved within one working day from the original request.

Security is maintained through a controlled Representatives of State. access system. Federal or private entities have access to the laboratory facility and records during laboratory normal business hours. must enter/exit through a central reception area. The receptionist keeps a date/time log. After work hours, the building is secured and linked to a commercial security agency. The security system is equipped with perimeter alarms, motion sensors, and speakers that monitor background sounds. Heat activated fire alarms are monitored by an outside A fire alarm also activates the security system. ATL SOP #30 describes the security and controlled access protocols.

2.3 EQUIPMENT AND INSTRUMENTATION

The laboratory is equipped with over \$2,000,000 of instrumentation, dedicated exclusively to the analysis of air samples. Much of the commercially available equipment is modified in-house in order to enhance performance in the areas of:

- overcoming challenging sampling problems;
- analyzing difficult matrices;
- achieving greater sensitivity.

A staff of design engineers and a 1,000 square feet fabrication shop is maintained by ATL in order to build, test, and service the custom equipment. A facilities map and equipment list can be found in Exhibit 2.2 and Tables 2.1 and 2.2.

Exhibit 2.2. Facilities Map

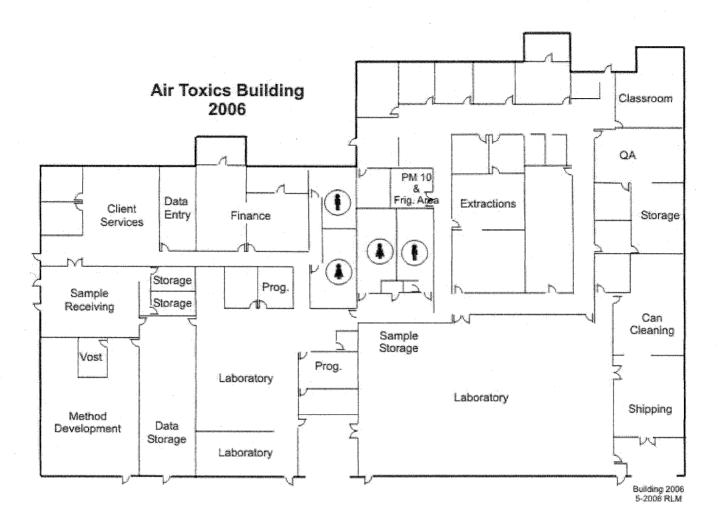


Table 2.1. Laboratory Instrumentation and Equipment

#	Description	Acquired
15	Agilent 5973 GC/MSD	1999 - 2005
4	Agilent 5975 GC/MSD	2005
1	Leco Time of Flight MS	2004
1	Markes Autosampler	2005
1	Hewlett-Packard 5980 GC/ECD/ECD	1995
1	Hewlett-Packard 5980 GC/SCD	1994
2	Hewlett-Packard 5890 GC/FID	1989, 1993
1	Hewlett-Packard 5890 GC/TCD/FID	1989
2	Hewlett-Packard 6890 GC/PID/FID	1999, 2002
1	Hewlett-Packard 5890 GC/TCD/ECD	1993
1	Sequoia-Turner Spectrophotometer	1994
1	SIS Shortpath Thermal Desorber	1998
1	Hewlett-Packard 1050 Gradient HPLC	1994
2	Air Toxics Canister Autosampler	1999
2	Air Toxics Custom Sorbent Tube Desorption	1992, 2002
	Unit	`
1	Air Toxics Permeation calibration system	1998
3	Canister pressurization stations	various
40	Soxhlets Extractors	various
1	Automated Canister CART Cleaning Station	1999
2	Automatic Solvent Extraction System	2003
8	Custom Convectron Pressure/Vacuum Canister Cleaning Manifold	various

Table 2.2. Sampling Media

Description	Quantity
Air sampling canisters	
6-Liter Summa canister	1900
1-Liter Summa canister	1200
PAC250 Summa Canister	260
High Pressure Sample Cylinder	30
Flow Controllers for air sampling canisters	800
24-hour flow controllers for canisters	500
Vacuum gauges	200
Tedlar bags: 1, 3, 10 liter	In inventory
MM5 air sampling traps	20
Midget impingers	30
VOST tubes kept in inventory	50 pair
TO-13 PUF/XAD and TO-4/TO-10 air sampling	200
cartridges	
TO-17 CarboTrap 300 air sampling tubes	150

3.0 QUALITY PROGRAM PLAN

Air Toxics Limited maintains comprehensive Quality Assurance programs to ensure that analyses are being conducted according to prescribed analytical methodology, and are within project specific QAPP requirements. The program is an integrated system of activities involving planning, quality assessment, quality control, reporting, and quality improvement. The basic elements of this program include:

- **DOCUMENTING** procedures, method requirements, and project requirements
- Organizing, monitoring, and leading TRAINING programs on quality related issues
- ASSESSING adherence to requirements, including maintenance of a system which documents, tracks, and provides closure when corrective actions are necessary
- Formally COMMUNICATING results of those assessments to laboratory management

These critical elements of the Quality Plan are described in detail in the following sections.

3.1 DOCUMENTING

3.1.1 The Quality Assurance Manual

The Quality Assurance Manual describes the major programs or systems by which the laboratory provides data of known and predictable quality. The QA Manager and the President are responsible for the content, accuracy and completeness of the Manual. The Manual must comply with all State and Federal requirements for those programs in which the laboratory maintains accreditation. The Quality Assurance Manual is a required reading for all laboratory staff and everyone must comply with the procedures documented

as a condition of continued employment. Each staff person documents in his/her training record that the latest revision Manual has been read and understood. The Department Manager and/or the Team Leader assess the accuracy and completeness of the documentation annually at the time of the employee's performance review. Missing or incomplete documentation is noted in the performance review.

The Quality Assurance Manual is reviewed and updated annually. All personnel are required to document reading the latest version in their training record.

3.1.2 Standard Operating Procedures/Methods Manual

The laboratory procedures used at ATL are documented in method-specific standard procedures (SOPs). operating These procedures are based on standard EPA or ASTM methodology whenever possible. The SOPs contain all necessary OC parameters, acceptance criteria, and directions for corrective action measures. The SOPs govern the laboratory response to results that are acceptance limits outside and address anticipated problems with associated recommended corrective action to eliminate the problem or further occurrences of the problem. SOPs also specify the type of written records (typically Corrective Action Requests known as CARs) necessary to fully document anticipated as well as unanticipated problems. The SOPs are maintained in numerical order in binders, which also serve as the laboratories Methods Manual. The SOPs address the following (where applicable):

- Identification of the test method
- Applicable matrix or matrices
- Limit of Detection (MDL)
- Method reporting limits
- Scope and application (includes target analytes)
- Summary of the method

- Table of significant variances from the method
- Definitions and interferences
- Safety
- Equipment and supplies
- Reagents and calibration standards
- Sample preservation and storage requirements
- Quality control
- Calibration, validation, and standardization
- Procedures
- Data analysis and calculations
- Method performance objectives
- Pollution prevention (if applicable)
- Data review and acceptance QC criteria
- Corrective Action for out of control data
- Waste management (if applicable)
- Method identifier and references
- Any relevant tables, flow charts or diagrams

The SOPs are written by the Department Manager, the Team/Task Leader or an experienced Scientist and are reviewed annually for technical accuracy and adherence to general OA/OC protocols. The SOP is signed and dated by the author, then is submitted for technical review, OA review, and final review by the laboratory director. Each method SOP contains a detailed table of all modifications taken against the actual reference method. Modifications and/or additions to the SOP are similarly reviewed and signed. Each hard copy SOP carries a unique revision number, control copy number, and date of generation. The SOPs are treated as confidential and proprietary and are maintained under the authority of the QA department. The original is kept in the QA department and extra copies in various laboratory sections as needed. versions of the SOPs are stored on a secured network drive, which only the OA Department SOP summaries that include can access. analyte lists, reporting limits, QC criteria and current variances to published methods are available to clients in .pdf form as the ATL

Methods Manual Summary. Copies of SOPs are made available to State and Federal accreditation and regulatory entities.

Current SOPs are stored electronically in a secured read-only database to allow review online by laboratory personnel. Whenever an SOP is updated and implemented, it appears in the electronic database. The QA personnel inform the Department Managers and the Team Leaders of the availability of the revised SOP. The Department Managers or the Team Leaders inform team members who then access the SOP from the laboratory network in SOP. to read the Once the Scientist/Analyst reads the SOP, he/she logs the date in the SOP tracking software, or signs and dates a copy of the title page from the hard copy stored in the laboratory. This documentation is then filed in his/her training record. The Department Manager and/or the Team Leader assess the accuracy and completeness of the documentation annually at the time of the employee's performance review. Missing or incomplete documentation is noted in the employee's performance review.

A comprehensive list of ATL's SOPs can be found in Appendix B.

3.1.3 Revisions to SOPs

The revision number of the referenced method is noted in the method-specific SOPs. The protocols and deviations are specific to that revision number. Air Toxics does not operate under more than one version of a referenced method at any time. The specific protocol used for analysis can be tracked using the effective date noted on the front page of the SOP.

Each SOP update is identified by a unique revision number. As with referenced method revisions, only one revision of an SOP is used in the laboratory at any one time. A complete description of ATL's system for writing and updating SOPs can be found in ATL's SOP #46.

3.1.4 Documenting Method Specific Deviations

Most air methods were not written as definitive and all have a strong performance based component to them. It is not unusual for the lab to have to design and create sampling interfaces or moisture control devices or to add additional quality assurance requirements to the methods in order to meet program more stringent project or requirements. Any variances to referenced methods are summarized in tabular form in the laboratory SOP. Signatures of the Laboratory Director and QA Manager on the front page of each SOP indicate review and approval of these variances. A copy of the method modifications table from the SOP appears in the Laboratory Narrative section of the comprehensive validation package or standard final report (Table 3.1). The QA team maintains and updates the templates used to create the Laboratory Narrative section of each work order. Each template has a revision date to ensure that only the most recent SOP table appears in the Laboratory Narrative.

On occasion, the need arises to change some aspect of an established SOP to accommodate enhancements to either the equipment or the method. The bench chemist documents the request for the change and the reasons behind it in the 'Request for Technical Change' form (Exhibit 3.1). The form is routed through the Department Manager and Team Leader with approval for the change noted by signature of the Laboratory Director and Technical Director. The form also identifies SOP's which established should revised/amended to incorporate the changes made. The forms are serialized in order to track progress and implementation. SOP Amendment forms are also used to reflect changes that are made to SOPs prior to a new revision.

Table 3.1. Example Method Modification Table

Requirement	EPA Method TO-3	ATL Modifications
Preparation of Standards	Levels achieved through	Levels achieved through loading
	dilution of gas mixture	various volumes of the gas mixture
Initial Calibration	4-point calibration using a	5-point calibration using average
Calculation	linear regression model	Response Factor
Initial Calibration Frequency	Weekly	When daily calibration standard
		recovery is outside $75 - 125 \%$, or
		upon significant changes to
		procedure or instrumentation
Daily Calibration Standard	Prior to sample analysis	Prior to sample analysis
Frequency	and every 4 - 6 hrs	·
Minimum Detection Limit	Calculated using the equation	40 CFR Pt. 136 App. B
(MDL)	DL = A+3.3S, where A is	_
	intercept of calibration line	
	and S is the standard deviation	
	of at least 3 reps of low level	
	standard	
Moisture Control	Nafion system	Sorbent system

Exhibit 3.1. Example Technical Change Request Form

Request for Technical	Change Form	No. <u>A-001</u>
This form is to be used	to propose any technical changes from	ATL's Standard Operating Procedures.
Initiated By:	Date:	
I. Description of Prop	osed Change:	
II. Reasons for Propos	sed Change:	
	Program April 198	
III. Method/Instrume	ents Affected:	
		(continued)

Exhibit 3.1. Example Technical Change Request Form Continued

IV. Approvals:	Danion off. Daniple Teemine		
Laboratory Director	Approval:		
		Linda L. Freeman	Date
Technical Director	Approval:	Signature	Date
Department Manag	er Approval:	The second secon	
V. Changes in AT	LAS Database	Signature	Date
Valid Values Table	(s)/Analyte Lists changed by:		
VI. Affected SOPs		Signature	Date
SOP/Rev #:		Revised/Amended by:	
			Initials
SOP/Rev #:	Date Revised/Amended: _	Revised/Amended by:	Initials
SOP/Rev #:	Date Revised/Amended:	Revised/Amended by:	
Initials:	Date:		
VIII. Notifications			e ani sis il se suo
QA Manager:			
		Signature	Date
Client Services Tea	m Leader (as required):	Signature	Date
Team Leader (as re	auired);	Organiture .	Date
		Signature	Date
Team Leader (as re	quired):	Signature	
Team Leader (as re	quired):		
		Signature	Date
Feam Leader (as required):		Signature	Date
Date Implemented			
This change to ATI	's Standard Operating Procedures	has been implemented by:	
QA Signa	ture Date		
@Air Toxics Ltd.		Revision Da	ate: 10/05

@Air Toxics Ltd.

3.1.5 Documenting Project Specific Deviations

Project specific OAPPs are reviewed by the Project Chemist (or designee) or a Technical Director. Team Leaders and key Scientists may review these documents as well. The laboratory may also take variances against method criteria established in project specific SOWs or QAPPs. The Project Chemist (or designee) reviews the project specific criteria during project proposal and notes any variances from standard laboratory SOP in a table. A variance table (Table 3.2) is then incorporated into the bid proposal for review and acceptance by the prospective client. The client notes acceptance by signature or initial and date in the designated field of the table. The project specific variance table is stored on a secured network drive following approval. A Project Profile (Exhibit 3.2) is initiated at the same time. A summary of the analytical requirements which differ from ATL's relevant SOP is documented in a Project Requirement Table and included in each Work Order folder. The Project Profile may also be accessed through the ATLAS database. The Project Chemist (or designee) or a Client Service Representative is responsible for noting the location of the Project Requirement Table in the Project Profile. Finally, the included variance table is Comprehensive Validation Package (CVP). The project specific QAPPs are maintained in file cabinets located in the Client Services Department.

Table 3.2. Example Project Specific Variance Table

sow	ATL SOP	VARIANCE APPROVAL*
Method 2720C Fixed Gases	ASTM D1945 Fixed Gases	
RDLs determined upon receipt of lab MDLs	Standard lab RDLs of 10 ppmv	
Field blank can one per batch	Not provided	
Canister released 90 days past reporting	Canisters released 30 days past day of sampling	
Initial multilevel calibration %RSD <30% for all compounds	Will incorporate for this project	No variance needed
72 hour retention time study	±0.06 minutes standard SW- 846	
Calibration verification daily with all analytes ±25% expected value	All compounds within 15%; Spike concentration is 25 ppbv	
Accuracy/precision study per analyst with project limits	Per analyst once every 12 months using ATL standard limits only	
LCS once per 5 point, ±25% difference for all compounds	All compounds within ±15%; Spike concentration is 25 ppby	

*NOTE: Each variance needs to be approved by the client's initial and date. An initialed copy of this variance table will appear in the Comprehensive Validation Package.

Revised: 05/22/01

3.2 TRAINING

3.2.1 Team Training

ATL laboratory staff members have sufficient education, training, and technical knowledge to perform their assigned duties. Each team has both experienced and in-training staff members. Those in training work under the supervision of a more experienced peer who is typically the lead Scientist assigned to that area.

Training of laboratory staff in analysis consists of three developmental stages:

• STAGE I. Introduction

Initial instruction by the analytical team leader or an experienced staff member concerning basic elements of the method and brief overview of instrumentation. Applicable SOPs and methods are read. During this time, the trainee is an observer.

• STAGE II. Training

Periods of close contact and direct supervision by an experienced staff member. During this time, which may last for several weeks, the analyst performs tasks independently. All aspects of his/her work are reviewed by the supervisor or experienced staff member.

• <u>STAGE III. Advanced Operation</u> Independent work with data review by the analytical team leader or a designated peer.

The final step in the training process allows the bench Analyst to document competency by analyzing four consecutive Laboratory Control Samples which is documented by a Demonstration of Capability Form (see Exhibit 3.5). A Continuing Demonstration of Capability must be made on an annual basis and documented by a Continued Method Proficiency Form (see Exhibit 3.6). Personnel who perform on odd shifts and do not commonly spec out instrumentation may substitute a duplicate analysis with acceptable precision (%RPD) for the four Laboratory

Control Sample analyses when fulfilling the Continuing Demonstration of Capability.

The QA team ensures that training of each member of the technical staff is complete, documented, and up to date. Exhibit 3.4 is an example of a Training Record form. It is the responsibility of the employee to keep his/her record current. An Analyst's/Scientist's training is considered current if the training record contains evidence that the employee has:

Training Record Checklist

- read the current version of the QA Manual
- completed laboratory training record
- completed necessary internal or external training classes
- completed the training class on ethical responsibilities
- read and understood the current version of relevant SOPs
- demonstrated initial proficiency in the methods by acceptable performance on four Laboratory Control Samples; proficiency is measured by accuracy and precision
- demonstrated continued proficiency in the methods by acceptable performance on four Laboratory Control Samples or duplicate analysis with RPD ≤ 25% between two analysts; proficiency is measured by accuracy and precision

The Department Manager and/or Team Leader review the training record on a yearly basis during an employee's annual performance review. Deficiencies in the training record are documented and returned for correction (see Exhibit 3.7). Consistent failure to maintain updated training records is noted in the performance review and effects the employees overall job rating.

A series of classes taught by in-house experts are offered throughout the year. Topics range from in depth technical aspects of the instrumentation to an overview of selected methods. Exhibit 3.8 contains a sample of internal courses that have been offered. The list of courses is subject to change on a yearly basis as a function of availability of instructors. Course attendance is mandatory for topics specifically related to an employee's job function. The QA department and management teams determine which courses Course attendees may be are mandatory. tested to ensure that they have achieved an acceptable understanding of the material presented. Completion of courses is documented in the employee's training record.

3.2.2 External Training

External training courses offered by software experts, instrument manufacturers, or other recognized experts in analytical instrumentation and/or analysis are attended by ATL employees. The course description, dates offered and record of attendance are kept in the employee's training records. The company maintains a budget for external training classes and higher education.

3.2.3 Quality Training

All new Air Toxics Limited employees are required to attend the Quality Assurance Orientation course. Completion of the course is documented in the employee's training record. The course outline includes:

- Introduction to QA and Laboratory Nomenclature
- How to Use CARs
- Definitions of SOPs, LQAP and QAPP
- Training Documentation
- Ethics I (Overview)
- Ethics II (Annual)

3.2.4 Health and Safety Training

Laboratory staff may, on occasion, be exposed to the handling of flammable solvents, compressed gases or toxic calibration standards. There are four to six staff members comprising the Safety Committee. Some members are 40 hour OSHA trained and respirator fitted. Education in the safe handling and disposal of these materials is accomplished as follows:

- Each new employee is given a safety tour of the facility within the first two weeks of employment. Documentation of this orientation appears in the employee's training record.
- The safety committee meets quarterly (or more frequently if needed) to discuss safety concerns and ways of improving safety in the work place.
- The safety committee schedules on going safety training throughout the year.
- If special precautions must be taken to perform a method, a safety section is included in the method SOP which discusses protocols and other measures for risk reduction through exposure prevention.
- ATL maintains Material Safety Data Sheets (MSDS) for each chemical used on-site. The MSDS are accessible to all personnel in the library area.
- ATL has access to MSDSs on the Internet through its vendor, VWR.

The safety committee staff members are assigned to duties including hazardous waste disposal, incident or spill management, staff training, Chemical Hygiene Plan review, and leading the safety committee.

3.3 ASSESSING ADHERENCE AND COMMUNICATING FINDINGS

The QA team plays a key role in establishing quality policy and protocols. The OA Department ensures that the established guidelines are followed through various quality control programs, which are designed to detect non-compliance or departure from protocol. Each quality control program includes documentation of the assessment feedback process and timely to the management and staff involved.

3.3.1 Data Review

The QA team reviews Work Orders which the client has requested 100% QA review. Deficiencies noted during review are documented and communicated to the staff involved.

QA REVIEW CHECKLIST

- Assessing accuracy and completeness of the laboratory narrative
- Assessing Analysis/Reporting vs. Project Profile/SOP requirements
- Documentation of any corrective actions
- Documentation of unusual circumstances
- · Verification of the QC meeting criteria
- Verification of sample dilution factors
- Appropriate peak integration and documentation for manual integration
- · Verification of appropriate data flags
- Verification of sample id's vs. COC
- Verification of reporting list, units and report header information
- · Verification of sample holding time
- Verification of adherence to analytical sequence clock times
- Verification of the appropriate Initial Calibration
- Verification of sample reporting limits
- Manual verification of one sample result from raw area counts
- Assessing accuracy and completeness of the Client Lumen report (if applicable)

The QA reviewer will look both for appropriate as well as inappropriate laboratory practices. Inappropriate practices are those which fall outside established laboratory

SOPs. If inappropriate practices are suspected, the QA reviewer will verify the result with the QA Manager and the Department Manager and/or Team Leader, initiate a Corrective Action Request, and if necessary, form a committee consisting of but not limited to the Department Manager and/or Team Leader, Technical Director, and Laboratory Director. Most Corrective Action Requests may be traced to human error. Oversights of this nature are simply documented and feedback is given to the Analyst or Scientist involved.

On occasion, the committee may determine that the Corrective Action Request was not attributable to simple human error. for the non-conformance reasons appropriate action to be taken are discussed and implemented by this committee. Typical actions include retraining of the individual involved along with a remedial period of close monitoring by the Department Manager and/or Team Leader. The QA team or an approved peer reviews all data reported by the Analyst during the remedial period. The laboratory uses a three-strike rule with respect to noncompliance issues. New Analysts are rigorously trained to follow SOPs. This training lasts several months. Re-training is done if there is cause to suspect noncompliance. Secondary training typically lasts Any further evidence of non-30 days. compliance may result in termination.

Client Services Representatives create Project Profiles that specify 100% QA review. When samples are received the Sample Receiving Team will automatically add a QA review to a workorder if 100% frequency has been requested as per the profile.

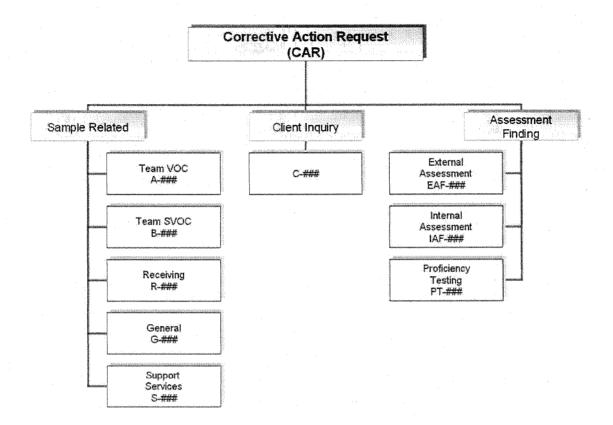
After QA review is complete the reviewer enters the review date in the sample tracking database. If a QA reviewer discovers an error, necessary corrections are made and the work order is reissued.

3.3.2 Corrective Action Program

The QA team manages the Corrective Action Program and maintains the Corrective Action tracking database. A Corrective Action Request (CAR) is initiated any time sample results are adversely affected by system noncompliance with established SOPs or program requirements, any time an internal or external assessment results in a finding, any time there is a failed proficiency evaluation sample, any time there is a failure quality system such that data quality is affected, and lastly, any time there is a customer inquiry into the laboratory's data quality and laboratory error is found (see Exhibit 3.2). This request is documented using one of ATL's eCAR forms - see Exhibit 3.9. A database is used to track the date of resolution, the necessity for a follow-up, and the date the follow-up action is completed.

Corrective Action Requests which require immediate resolution must be completed and finalized within 2 business days. All other CARs must be resolved within 30 business days. The status of corrective actions that have not yet been completely resolved (including follow-up actions), are discussed during the weekly OA Meetings. Whenever a customer raises an issue relating to data quality, the inquiry is documented in the Atlas Contacts database. A representative of the QA team reviews the data in question and investigates any systematic problem that may If results of the review and be evident. investigation merit corrective action a CAR will be initiated along with any necessary follow-up action.

Exhibit 3.2. Types of Corrective Action Request Forms



Examples of when the client inquiry CAR may be initiated include:

- Blind field duplicates that do not agree
- Field blanks that had contamination present
- Blind proficiency sample that did not meet accuracy objectives
- Sample splits that do not meet precision objectives
- Outlet sample results that were higher than inlet sample results
- Sample results that cannot be manually verified
- Sample results that do not meet program requirements

A portion of the CAR database is associated with sample receiving and analysis. Should a malfunction occur with a pending sample, the client is contacted prior to analysis to confirm if the analysis should continue. The CAR form documents the contact and resolution of the issue. Should the decision be made to proceed with analysis then any malfunction affecting data quality is detailed in the laboratory narrative. Instructions to proceed with analysis and narration of the affected results are documented in the CAR form. The Department Manager and/or Team/Task leader must review the CAR and determine if the error is isolated or systematic. After QA review, the CAR form is then filed with the Work Order as a permanent record of the nature of the problem and the resolution. A complete description of ATL's CAR system can be found in ATL's SOP #61.

3.3.3 QA Management Meetings

Each week the QA Manager leads a meeting with the President, the Department Managers, the analytical Team Leaders, the Client Services Team Leader, and the Support Services Team Leader. All other Managers and Directors are welcome to attend. These meetings are called to discuss the effectiveness of the quality systems, specific quality issues

that may have surfaced during the week, and to monitor progress with respect to open Corrective Action items. Agenda items are added and removed at the discretion of the QA Manager or QA staff.

The meeting is used as an interactive forum in which non-compliance issues are discussed with respect to the overall suitability of the quality system involved. Non-compliances are screened to see if the quality system itself is in need of a review or modification. If it is determined that a particular quality system needs to be designed or revised then the responsibility takes committee restructuring that system. The issue cannot be removed from the weekly agenda until the new system is in place. Minutes of each meeting are kept in a QA electronic form and in a binder in the QA office.

3.3.4 Conducting Internal Assessments

The QA team conducts internal assessments of all major production areas of the lab on a yearly basis. The production areas are separated into assessment modules by referenced methodology. Whenever possible audits are scheduled to occur after the yearly update and revision of the relevant SOP. Audits are composed of three events:

- Laboratory assessment based on the current SOP by the QA Team
- Circulation of the assessment report and issuing of any necessary corrective action forms
- Satisfactory response to audit findings

An assessment checklist is developed for each area by the QA Manager or designated staff. The checklist contains general, method specific, and SOP specific practices (Exhibit 3.10). The assessment process addresses whether or not quality systems (e.g., adherence to the current revision of the SOP,

proper and complete documentation practices etc.) are in place and understood. Health and safety issues are also covered.

Results of the assessment are summarized in the checklist that serves as the basis of the report. Findings that are determined to be in need of Corrective Action are processed through the standard CAR program. If findings imply that there has been a significant impact on the data, the report will be corrected and reissued to the client. Copies of the internal assessment report are circulated to the Department Managers and Team Leaders and other members of ATL management team.

3.4 COMMUNICATING WITH MANAGEMENT

Results of the QA assessments are documented in a **Quarterly QA Status Report** that summarizes the numbers and types of CARs produced, the status of any outstanding CAR, results from proficiency testing samples, any internal and external assessment finding, a summary of customer inquiries received, and any general QA issue. This report is distributed to all Directors, Managers, and Team Leaders.

Exhibit 3.3. Example ATL Project Profile

Project Profile

Project Name			P	roject Nu	mber		P.O.#		
Big Landfill									
Project Descrip	ition								
Project Requirem	ent Table: O:\\	/ariances\2	00310	303-0010	C				
	Report to A	ddress				Bill to Add	ress		
Name:	Mr. John Jon					Mr. John Jo	FIRE		,
Company:	Average Eng	ineering Fir	m			Average En	gineering Firm		
Address:	1234 Anystre	et Avenue				1234 Anystr	reet Avenue		
						Your Town	IA 50841	and the stay of th	
City:	Your Town				Done:	Υ	QAPP on	File?	Υ
State/Zip:	IA 50841				CS Rep:	DD	Penalties1	,	N
Phone/Fax:	641-987-654				ProjectID:		24 Hour C	ock?	Y
Email:	641-234-567 jj@aef.com	8 (Fax)			Variance;	Yes	Charge Fo	Shipping?	N
		****		:					
Inalysis(es):	Repo	rting List:				Units	TAT	Price Sur	_
Modified TO-15	Modi	fied TO-15				ppbv	10 Day	,00 No	one
A/QC:					,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,				
Reporting List				Dups	CCV	LCS			
Modified TO-15		······································		10%	10%	1/ANB			
viedia:					Medi	a Price:			
Liter Summa C	anister					.00			
Deliverables:		Done: F	Price:	Price is:		Due:	Send VIA:		
Standard ATL R	eport		.00	Per WO	10 Wo	rking Days			
Client Specific D	•		nn	Per WO		- '	Mail		
Jacin Opeune L	ZISK I OIIIIai	<u> </u>							······································
Notes to Receiv	ring:								
Notes to Lab:									
Notes to Report	tina:								
irores to ireholi	9*							r	
							1940		
Miscellaneous	tems:					Price:			•

Exhibit 3.4. Example Laboratory Training Record LABORATORY TRAINING RECORD

EMPLOYEE: MET	ГНОD:	
The normal training program consists of	three developmental stages. Indicate the dates compl	ete
next to each stage. You should always	progress from one stage to the next in sequential or g with associated precision data is submitted to the Qua	de
STAGE I. Introduction		
concerning the basic elements instrumentation. Activities inclu	the Team Leader or a more experienced analyst of the method and a brief overview of the ude, reading the applicable SOP(s), reading the apples. During this period of time, the trainee is an brief.	
Date Completed:	Trainer:	
STAGE II. Training		
more experienced analyst. Ac Calibrations, routine maintenance	with and one-on-one training by the Team Leader or a ctivities include: performing Initial and Continuing and data reporting. During this time, which may last erforms tasks independently as assigned, but with all by the trainer.	5
Date Completed:	Trainer:	
STAGE III. Advanced Operation		
passed into the normal laboratory above, but the trainee acts indep	operates completely independently with his/her work review cycle. Activities include those listed in Stage II endently. An example of completion of this stage is analyzed and meet the method accuracy and precision attitation page and/or summary).	[}
EMPLOYEE:	DATE:	
TEAM LEADER:	DATE:	
QUALITY ASSURANCE:	DATE:	

Revised: 9/9/02

Exhibit 3.5. Demonstration of Capability Form

Date C	ertified:			
	tory Name:	Air Toxics Limited		
Addres	ss:	180 Blue Ravine Road, #B		
		Folsom, CA 95630		
Matrix	: <u>Air</u>			
	d Name:		-	
		sion:		
File Nu	ımbers:			
We, the	e undersigned, C	ERTIFY that:		
		Analyst's na	ame	
1.	the analyses of	entified above, using the cited test samples under the National Environmentation of Capability.		
2.	The test metho	d(s) was performed by the analys	t identified on this certificate.	
3.	A copy of the on site.	test method(s) and the laboratory	specific SOPs are available	for all personnel
4.	The data associated explanatory (1)	iated with the demonstration cap	pability are true, accurate, co	mplete and self-
5.	these analyses	ncluding a copy of this certificati have been retained at the facilit available for review by authorize	y, and that the associated infe	
Heidi (C. Hayes			
	chnical Director	Signature	Date	e .
	ie Levesque	~	D .	
Quality	y Assurance Mar	nager Signature	Date	2
This ce	ertification form	must be completed each time a c	apability study is completed.	
(1)	True:	consistent with supporting	data	
	Accurate:	11 0	practices consistent with	sound scientific
		principles and practices.	·	
	Complete:		ipporting performance testing	•
		natory: data properly labeled and no additional explanation		clear and require

Revised: 08/05

Date R	e-Certified:		
Labora	tory Name:	Air Toxics Limited	
Addres	s:	180 Blue Ravine Road, #B	
		Folsom, CA 95630	
Matrix:	: <u>Air</u>		
Method	l Name:		
SOP N	umber and Rev	sion:	
We, the	e undersigned, (CERTIFY that:	
		Analyst's name	
1.	the analyses of	entified above, using the cited test method(f samples under the National Environmenta Continued Method Proficiency.	
2.	The test method	od(s) was performed by the analyst identifie	ed on this certificate.
3.	A copy of the on site.	test method(s) and the laboratory specific	SOPs are available for all personnel
4.	The data associated self-explanator	ciated with the Continued Method Proficiery (1).	ncy are true, accurate, complete and
5.	these analyses	ncluding a copy of this certification form) have been retained at the facility, and the available for review by authorized assessor	at the associated information is well
Haidi (C. Hayes		
	chnical Director	Signature	Date
, 1, 100		Signature	. *
Melani	e Levesque		
	Assurance Ma	nager Signature	Date
This ce	rtification form	must be completed each time a capability	study is completed.
(1)	True:	consistent with supporting data	
· · · · · ·	Accurate:	based on good laboratory practice principles and practices.	es consistent with sound scientific
	Complete:		nerformance testing
		natory: data properly labeled and stored so no additional explanation	

Revised: 02/06

Exhibit 3.7. Example Training Record Review Check Sheet

Reviewed By:

TRAINING RECORD REVIEW CHECK SHEET **Employee: Items Present** Resume Documentation of Reading current version of the LQAP Documentation of Ethics Training Documentation of Safety Training Documentation of reading current version of applicable SOP(s)/Methods Yellow Initial Training Record(s)/"Demonstration of Capability" form(s) Proficiency Data (Initial and Continuing) if applicable Documentation of Classes/Continuing Education Courses Yellow Training Record(s) "Date Completed" and "Trainer" filled out for each stage Employee Signature Team Leader Signature **Quality Assurance Signature Initial Proficiency Data** Four replicate LCSs (analyzed on the same day or on separate days Meets method criteria for Accuracy and Precision Date and initials of reviewer on Proficiency Data NELAP "Demonstration of Capability" form completed **Continued Proficiency Data** Four replicate LCSs (analyzed on the same day or on separate days), ICAL with Second Source Check, MDL Study, or Duplicate Analysis (%RPD ≤ 25% between two Analysts) Performed within a year of review date Meets method criteria for Accuracy and Precision Date and initials of reviewer on Proficiency Data Current NELAP "Demonstration of Capability" form COMMENTS: (NOTATION OF MISSING ITEMS, INCORRECTLY COMPLETED FORMS, ORGANIZATION, ETC.)

Date:

Revised: 06/14/02

Exhibit 3.8. Examples of Internal Training Courses (Subject to Change)

Gas Chromatography
Column Selection, Care & Maintenance
Detectors – Overview of Team VOC & SVOC
The mass spec detector - specific training
GC Parameters
Injection Techniques
Proper Use of Fittings and Gas Traps
110001
Quality Assurance
Introduction to QA and Lab Nomenclature (2
sessions)
Calibration and Analysis
Standards Preparation and Documentation
Syringe & Dilution Techniques
Proper Log Book Protocol
Canister Pressurization
Correct Operation of Manual Interface
NOAH – Overview
The ATL Way
Bidding through Invoicing, the Process
Training Records - How to set up & maintain
Health & Safety Training
Integrity Training – Ethical Laboratory Practices
Computer Systems and Security
On the Job Team Specific courses
Detectors – Specific Training (PID/FID, ECD)
Hydrocarbon Profiling and NMOC
NIOSH Overview – 2456 & 5515
EDD Production
Understanding Analysis of Atmospheric Gases
Understanding Analysis by TO-3
Understanding Analysis by TO-13/8270
Overview of Sulfur Analysis & the SCD
Overview of Headspace (RSK-175) analysis
Understanding Analysis by TO-14A/15
Understanding VOST Analysis
Power Point
Ethical and Defensible Manual Integration

Exhibit 3.9. Example eCAR Form

Corrective Action Report No.____ Initiated By: Date: Team Leader: Date: CSR: Date: Date Closed: QA Dept: Team(s) affected: Method(s) affected: Work order(s) affected: Project Profile ID(s): Sample(s) affected: CSR: Describe the nonconformity and its cause: **Describe the Corrective Actions:** Client Notification:

By:

Date:

Client Instructions:

Person Notified:

Person Notified:	By:	Date:
•		
Additional Corrective Actio	ns:	
MEMORIAN PARAMETER AND		
Date Implemented:		
FT 38 4 4 56 76 7	0	
Follow-up Action Necessary	?	
Describe Follow-up Action:		
Date Follow-up Action Com	pleted:	
Completed By:		
Compicted by.		

Exhibit 3.10. Example Internal Audit Checklist

EPA Method SW5041A/8260B/TO-17 Technical & Quality Audit Checklist

Category OK? Not OK Comment CAR? Instrument Operation continued How long and at what flow rate are TO-17 samples dry purged prior to analysis? Blanks? What temperature is used for TO-17 desorption? Standard Preparation What are the concentrations of the liquid working standards? What compounds are contained in the working gas standard? What are the calibration levels? What is the concentration of the Internal Standard/Surrogate mix? What volume is loaded? Calibration and Quality Control How often is a BFB Tune Check required? How is the ion ratio abundance checked? What are the criteria for Internal Standards in the daily CCV? What are the criteria for Internal Standards in Lab Blanks, Samples and all non-CCV QC checks? What is corrective action is taken when Internal Standard area counts do not pass ion the Lab Blank? What are the SPCC and CCC compounds and what is the criterion (RRF, %RSD, %D) for acceptability (ICAL and CCV)? What is the %RSD and %D criterion for the non-CCC compounds? If surrogate recovery is outside of control limits, what is the corrective action (VOST tubes and condensates)?

2

Exhibit 3.10. Example Internal Audit Checklist Continued EPA Method SW5041A/8260B/TO-17 Technical & Quality Audit Checklist

4

Category	OK?	Not OK	Comment	CAR?
Sample Analysis continued				
If recovery of Benzene-d6 is				3
not between 50-150%, is the				blingeren
dilution reanalyzed (provided				dimension
sufficient sample volume		-		-
remains)?				****
How is transfer efficiency from				
tube to Tedlar bag measured			· ·	- :
when dilutions are required?				
How is the initial load volume				
for a bag dilution determined?				war na
How is the dilution factor for	<u> </u>			
this analysis calculated?				and the second s
How is a plugged needle	1			
discovered during TO-17				approximate and the second
analysis?		-		and the second s
Document Control		1. N. W. W. W. W. (1971)		
How do you document				
Standard Preparation? Do you				incompany
know where the concentration				
calculation is located?				Commence of the Commence of th
Demonstrate the use of this			no managament de la companya de la c	-
calculation.		****		and the second s
How are prepared standards				
labeled?		Salation School	/	
Where are Standard				
certification documents?				
How do you document chain of				:
custody in the cage, and how				
do you indicate that a sample		en e		
has been discarded?				
Instrument Maintenance				
How often is the vacuum				-
gauge checked for potential				
system leaks?		1		
How often is the septum on the				
GC injection port replaced?				2
How often is the septum on the				a de la companya de l
valve syringe interface	No.		es.	reservation
changed (13.2.3)?				
What are the reasons (aside				and the state of t
from breakage) for changing			- in the second	ř.
the chromatographic column?	· ·			

4.0 QUALITY OBJECTIVES

The primary objective of the QA Program is to ensure that the laboratory is producing data that meet the laboratory's standard acceptance criteria for each method. Acceptance criteria from project-specific QAPPs are also used when required.

The laboratory's standard acceptance criteria and the sources of those criteria are specified in Sections 6.0-6.18 of this Quality Manual. Definitions of parameters used to assess the quality of the data are defined below.

4.1 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY

4.1.1 Precision

Precision measures the reproducibility of measurements. Analytical precision is the agreement among duplicate (two) or replicate (more than two) analyses of the same sample. The acceptance for precision is determined using the relative percent difference (RPD) between the duplicate sample results. The %RSD (relative standard deviation) is used to document precision of linearity for the initial calibrations. In addition, %RSD measures precision for triplicate analyses points in Method 25C. The formula for the RPD and RSD calculations are provided in Exhibit 4.1.

Field duplicate samples represent total precision, the reproducibility associated

with the entire sampling, and analysis process. However, the identification of field duplicate samples are typically not known to the laboratory, and therefore not specifically evaluated by the laboratory's QA department.

4.1.2 Accuracy

Accuracy measures correctness and includes components of random error (variability due to imprecision) systemic error. Analytical accuracy is measured by comparing the percent recovery of analytes spiked (as compared to the expected value) to pre-established accuracy limits (i.e., acceptance criteria). Any type of spiked sample measures accuracy. The formula for calculation of accuracy is included in Exhibit 4.1 as percent recovery (%R) from pure and sample matrices.

4.1.3 Representativeness

Representativeness is achieved through use of the standard analytical procedures described in this Quality Manual.

4.1.4 Completeness

Completeness is the percentage of data, which meets the established acceptance criteria referenced in Sections 6.0-6.18. ATL's goal is to achieve at least 95% completeness for both normal turn-around-time (TAT) and rush TAT data. Meeting the method specification outlined in each SOP prior to analyzing project samples is our means of achieving this goal.

4.1.5 Comparability

Comparability is the confidence with which one data set may be compared to another. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. Comparability is achieved by using standard analytical methods, reporting data in standard units, and using standard and comprehensive reporting formats.

4.2 LIMIT OF DETECTION, LIMIT OF QUANTITATION, AND INSTRUMENT CALIBRATION REQUIREMENTS

4.2.1 Limit of Detection

The Limit of Detection (LOD) is a statistically determined value (by Method Detection Limit per 40CFR Part 136 Appendix B). The LOD must be less than the Limit of Quantitation (LOQ). If the true concentration is below this value, the Each LOD analyte may not be detected. study is repeated at least once per twelvemonth period, when a new instrument is installed, when there is a major change in the analytical configuration such as column, detector, sample concentrator, sample loop size, etc. or when there is a major change in the extraction method such as solvent, extraction apparatus, clean-up procedure, etc.

All analytical constituents noted by methods in Section 6.0 are to be reported with a valid and current LOD, but in the case of special request compounds LODs are performed only when a client specifies it to be a project requirement. Special request compounds are reviewed by the Department Managers to determine the

cost to the laboratory for additional LOD analyses. The additional value added is then factored into the bid that is submitted to the prospective client.

4.2.2 Limit of Quantitation

ATL reports down to the Limit of Quantitation (formerly called the Practical Quantitation Limit or Reporting Limit) which is the lowest concentration contained in a linear calibration).

The LOQ represents a uniform value that can be accurately detected for any particular analyte on each instrument thereby providing consistency for samples analyzed on different instruments. The Reporting Limit is verified by the statistical and analytical LOD studies.

The acceptance criterion for the LOD study is a value of less than the LOQ. Corrective action including raising the LOQ may be performed if the statistically and analytically determined LOD does not meet the stated criterion.

4.2.3 Instrument Calibration

Analytical instruments are calibrated in accordance with the referenced analytical methods and internal SOPs. The acceptance criteria are summarized in Section 6.0. All specific target analytes are included in the initial and continuing calibrations.

If multi-point calibrations do not meet acceptance criteria stated in the relevant SOPs, an option to narrow the range of the curve either by eliminating the low point or the high point of the curve may be considered providing all project criteria Otherwise, the entire are still met. calibration curve is repeated. Reanalysis of any level of the multi-point calibration in order to meet OC acceptance criteria is not allowed unless there is evidence of an anomaly such as instrument malfunction improper load volume Documentation of the anomaly must accompany the raw data for the Initial Calibration. Elimination of any of the inner levels of the multi-point calibration in order to meet QC acceptance criteria is not allowed.

Records of instrument calibration and records that unambiguously trace the preparation of standards and their use in instrument calibration are maintained for 5 years. Calibration standards are traceable to standard materials.

A second source (or different lot) standard that contains all target compounds, as noted in the Section 6.0 tables, is analyzed after each initial curve to verify that the standards are correct and the calibration is accurate. The acceptance criteria for the independent source recoveries are presented in Section 6.0.

In the case of special request compounds, a second source analysis is performed only when a client specifies it to be a project requirement. Special request compounds are reviewed by the Department Managers to determine the cost to the laboratory for additional second source analyses. The additional value added is then factored into the bid that is submitted to the prospective client.

Analyte concentrations are determined primarily using the average RF from the initial multi-point calibration.

4.2.4 Retention Time Window

The techniques used to establish retention time windows for GC and HPLC analyses vary by method, based on the class of compounds targeted, as well as the instrument specifications (e.g., column type, etc.). Protocol for establishing retention time windows can be found in the method-specific SOPs.

4.3 ELEMENTS OF QUALITY CONTROL

The various types of QC samples are described below. The method specific laboratory QC sample frequency and acceptance criteria may be found in Section 6.0.

4.3.1 Analytical Batch Definition

For non-extractable methods, samples analyzed during a single 24-hour period along with associated matrix specific laboratory QC samples make up an analytical batch. At a minimum, any analytical batch will include a Laboratory Blank, CCV, LCS and an end check for non-GC/MS methods. Reporting of the batch QC samples varies according to project requirements. The number of field samples included in any one analytical batch is limited to 20.

In the case of samples that require extraction prior to analysis, the sample preparation process defines the batch. At a minimum, the sample preparation batch will include a Laboratory Blank and a Laboratory Control Sample (LCS). The maximum number of samples included

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within one preparation batch may not exceed 20 in one given day.

4.3.2 Continuing Calibration Verification (CCV)

A Continuing Calibration Verification (CCV) containing all analytes of concern is performed at the start of each day and, if required, at the start of every 12 or 24 hour clock for GC/MS analyses. GC and HPLC sample analyses are generally bracketed by opening and end check CCVs (TO-4A, TO-10A and PAMS methods excluded). Mid-batch CCVs are also analyzed as per individual SOP.

The concentration of the CCV is usually near the mid-level of the calibration. The CCV is analyzed at other concentrations within the working range at least once a quarter, or more frequently if specified in an SOP. If the CCV fails to meet the performance criteria then the test is repeated with the same standard (or optionally with a different preparation of the same calibration mix). If the second analysis fails criteria, maintenance should be performed and the test repeated a third time. If the system still fails the calibration verification, a new multi-point calibration curve is performed.

4.3.3 Laboratory Control Spike (LCS)

Each analytical or extraction batch contains at least one mid-level spike using a second source (or different lot) standard containing all (or in the case of extractable a subset of) the target analytes. In the case of non-extracted batches, the LCS is generally analyzed daily prior to sample analysis, but may also serve as an End Check standard. If the stated criteria are

not met, the system is checked and the standard reanalyzed. In the event that the criteria cannot be met, the instrument is recalibrated. In the case of extracted LCS, out-of-control recoveries result in data flags since samples cannot be re-extracted.

4.3.4 Internal Standard (IS)

For all GC/MS methods an IS blend is introduced as each standard, blank to monitor the stability of the analytical system. The internal standard acceptance criteria vary by method, but for all applicable analyses at ATL, if the internal standards for the blank do not pass the acceptance criteria, the system is inspected and the blank reanalyzed. Analyses are discontinued until the blank meets the internal standard criteria.

4.3.5 Surrogates

For GC/MS methods and some GC methods, the recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, and to provide a measure of recovery for every sample matrix. The surrogate recovery acceptance criteria vary by method, but for all applicable analyses at ATL, if the surrogate recoveries for the Laboratory Blank do not pass the acceptance criteria, the system is inspected and the blank is reanalyzed. Analyses are discontinued until the blank meets the surrogate recovery criteria.

In some extractable methods, surrogates are added prior to extraction to monitor the efficiency of the extraction process. If the surrogate recoveries are outside acceptance limits, reanalysis occurs. Reextraction of samples is not possible.

If the surrogate recoveries for a sample are outside the limits, the sample is reanalyzed unless obvious matrix interference is documented. If the surrogate recoveries are within limits in the reanalysis, the second analysis will be reported. If the surrogate recoveries are out of limits a second time, the initial analysis is reported with a narrative indicating that the acceptance criteria for surrogate recoveries are exceeded. Upon request, the data from the matrix effect confirmation analysis is provided to the client.

4.3.6 Laboratory Blank

A Laboratory Blank is analyzed after any applicable standards and prior to the analysis of project samples. A blank is also analyzed in the event saturation-level concentrations are incurred to demonstrate that contamination does not exist. For methods that involve an extraction, a Laboratory Blank is prepared with each set of no more than 20 samples per method per matrix.

The acceptance criterion for the Laboratory Blank is a result less than the Limit of Quantitation (Reporting Limit). Laboratory Blank is analyzed immediately after the LCS (nonextractable analysis) or the **CCV** (extractable analysis) to ensure that both the instrument and extraction process are free from contamination. When samples that are extracted together are analyzed on different analytical clocks, a solvent (instrument) blank is analyzed demonstrate that the instrument is free from contamination.

4.3.7 Laboratory Duplicate

Project samples are analyzed in duplicate on a minimum of 10% of the samples received. For some projects the required frequency is one duplicate analysis per analytical batch. The acceptance criteria for analytical reproducibility generally apply to analytes present at ≥ 5 times the Reporting Limit. If the noted criterion is exceeded, the sample is re-analyzed a third time. If acceptable reproducibility is still not obtained, the cause is investigated and the system is brought back to working order. If no problem is found on the system, the data is narrated to note the non-conforming event.

4.3.8 Matrix Spikes

Matrix spiking permanently alters the native concentrations of whole samples. Therefore, matrix spiking is performed only on samples, such as condensates, submitted as part of a sampling train or on waters submitted for Headspace analysis (RSK-175). When applicable, matrix and matrix duplicate spiking is performed using a subset of analytes. Recoveries and target reproducibility demonstrated values. which do not meet the acceptance criteria, are flagged and explained in the laboratory narrative.

4.3.9 Field QC Samples

Field blanks, field spikes, and field duplicates are generally treated as normal project samples by the laboratory. The exceptions include methods in which the laboratory at the direction of the client specifically prepares the sample media. To assure consistency it is recommended that

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certified summa canisters connected to a sampling tee be used for the collection of Field Duplicate samples.

4.4 QUALITY CONTROL PROCEDURES

4.4.1 Holding Times

All sample preparation and analysis are to be completed within the method-required holding times. The analytical holding time for a non-extractable method begins the day of sample collection. For extractable methods, the holding time is calculated from the day of sample collection for the extraction process and from the day the extraction process begins for the analytical process.

If holding times are exceeded, a CAR form (Section 3.3.2) is generated, the client is notified, and situation is narrated on the final report.

4.4.2 Confirmation

GC and HPLC methods do generally not perform quantitative confirmation for air sample analysis. The exception is for analysis of pesticides by SW-846 methodology, in which case, second column confirmation is completed within the method-required holding times.

4.4.3 Standard Materials

All neat and liquid standards used are traceable to the National Institute of Standards and Technology (NIST) and NIST traceable weights are used to verify balance calibration. Documentation from the manufacturers is maintained to verify each standard. Gaseous standards (which

are by nature unable to be quantified on a balance) are verified by accuracy documentation supplied by the manufacturer.

A second source (or different lot) standard is used to confirm the accuracy of primary source calibration standards. Ideally the second source is obtained from a vendor other than that of the primary standard. In the case of TO-14A/15 a reliable second source vendor may be difficult to find and therefore a different lot standard may be used for this purpose. These standards are used for the laboratory control samples as well. Non-standard and polar TO-14A/15 compounds may be prepared from neat standards. Second source standards for these compounds are either derived from a different vendor or from a different lot if only one vendor exists.

4.4.3.1 Liquid Standards

Liquid Standards are prepared by dilution from commercial sources. Stock solutions are purchased and stored in a separate refrigerator/freezer. Dilutions to working ranges are prepared using high purity solvents. Solvents are logged into the receiving logbook and the date of arrival is documented. Open solvent containers are stored in a vented, flammables cabinet.

4.4.3.2 Gas Standards

Gas standards are purchased from a commercial supplier and stored in vendor recommended cylinders using high purity regulators. Standards that are not available in certified blends from commercial suppliers are purchased in neat form. Neat materials are purchased with a purity of at least 96% whenever possible.

Certified gas blends are purchased at parts per million volume (ppmv) levels and diluted into the working range by transfer into 1.0 L or 6.0 L certified evacuated summa canister. The canister is then pressurized to 5.0 psi or 15.0 psi depending on the volume. Alternatively, a high purity flow controller is used to fill a conditioned Tedlar bag with a controlled volume of N2 or zero air. Neat liquid standards are transferred into the Tedlar bag by injection to achieve the desired The standard is given concentration. sufficient time for equilibration and then is transferred into a conditioned summa canister and pressurized appropriately to achieve the desired final concentration.

Concentration of the blend is determined using density based calculation:

$$ppbv = \frac{ng/MW}{vol(L)/24.45*}$$

* 24.45 is the molar volume of any gas at normalized pressure (1 atmosphere) and temperature (25°C), derived form the ideal gas law (PV = nRT), where R = the universal gas constant.

Once blended, the standard is transferred into a SummaTM canister for long-term storage and stability.

The preparation of working standards, in gaseous or liquid states, is documented in bound standard preparation logbooks. Each standard is given a unique identification number. Additional information including the solvent or standard lot number and stock standard concentration is noted. Each page is signed and dated by the analyst.

4.4.3.3 Reagent Water

The laboratory uses water to prepare moist Laboratory Blank canister samples, VOST condensate water blanks and water impinger blanks. The volume of water these required for purposes insignificant. As such, the laboratory relies on high purity HPLC grade bottle reagent water, which is subjected to a constant purge flow of Ultra High Purity Nitrogen. The water is purchase certified then supported by certifying and Laboratory Blank analyses.

4.4.4 Expiration Dates of Standards

4.4.4.1 Primary standards expire according to the manufacturer's expiration date. If the manufacturer does not assign an expiration date, a period of one year from the date of opening is applied. Expiration dates are noted on standard labels. Expired standard materials are either revalidated by comparison with unexpired independently prepared standards, or are discarded. The acceptance criterion for standards revalidation is documented in ATL SOP #33. The newer of the two standards is always used as the primary source.

Expiration dates for laboratory-prepared stock and diluted standards are no later than the expiration date of the stock solution or material.

All efforts are made to obtain the highest purity possible when purchasing neat chemical standards. The vast majority of neat standards are \geq 96% pure. The concentration of material purchased at less than 96% purity is corrected mathematically to assure that dilutions for working standards are accurate.

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Neat liquid standards are used until analysis by GC/FID indicates a purity of less than 96% (or less than the stated purity for the exceptions). The date of the purity check is noted on the neat standard vial.

Purity analysis is performed once per year as needed.

4.4.4.2 Secondary Standards

Secondary Standards are assigned based on the expiration date of the primary source standard (i.e., no later than), the compounds present, and container type. Typical expiration dates are presented in Exhibit 4.2.

Exhibit 4.1. Statistical Calculations

Statistic	Symbol	Formula	Definition	Uses
Mean	\bar{x}	$\frac{\begin{pmatrix} n \\ \Sigma & X_{i} \\ i=1 \end{pmatrix}}{n}$	Measure of central tendency	Used to determine average value of measurements
Standard Deviation	S	$\left(\frac{\sum (X_{i} - \overline{X})^{2}}{(n-1)}\right)^{\frac{1}{2}}$	Measure of relative scatter of the data	Used in calculating variation of measurements
Relative Standard Deviation	RSD	(S/X) x 100	Relative standard deviation, adjusts for magnitude of observations	Used to assess precision for replicate results
Percent Difference	%D	$\frac{X_1 - X_2}{X_1}$ x 100	Measure of the difference of 2 observations	Used to assess accuracy
Relative Percent Difference	RPD	$\left(\frac{(X_1 - X_2)}{(X_1 + X_2)/2}\right) \times 100$	Measure of variability that adjusts for the magnitude of observations	Used to assess total and analytical precision of duplicate measurements
Percent Recovery	%R	$\left(\frac{X_{\text{meas}}}{X_{\text{true}}}\right)$ x 100	Recovery of spiked compound in pure matrix	Used to assess accuracy
Percent Recovery	%R	value of value of spiked - unspiked sample sample x 100	Recovery of spiked compound in sample matrix	Used to assess matrix effects and total precision
Correlation Coefficient	r	see SW8000B section 7.5.3		Evaluation of "goodness of fit" of a regression line

X = Observation (concentration)

n = Number of observations

Exhibit 4.2. Expiration Dates

Gas Standards Prepared from Certified Cylinders

Compounds	Tedlar Bag Standard	Summa™ Canister
TO-14A/15 List of 41 compounds	3 days	3 months
BTEX/TPH	3 days	3 months
Sulfur Compounds > 3000 ppbv	7 days	NA
Sulfur Compounds < 3000 ppbv*	1 day	NA

Gas Standards Prepared from Neat Materials

Compounds	Tedlar Bag Standard	Summa™ Canister
TO-14A/15 Extra Compounds	3 days	6 months
Other Compounds	3 days	6 months

Liquid Manufacturer's Certified Mix and Single Component Standards

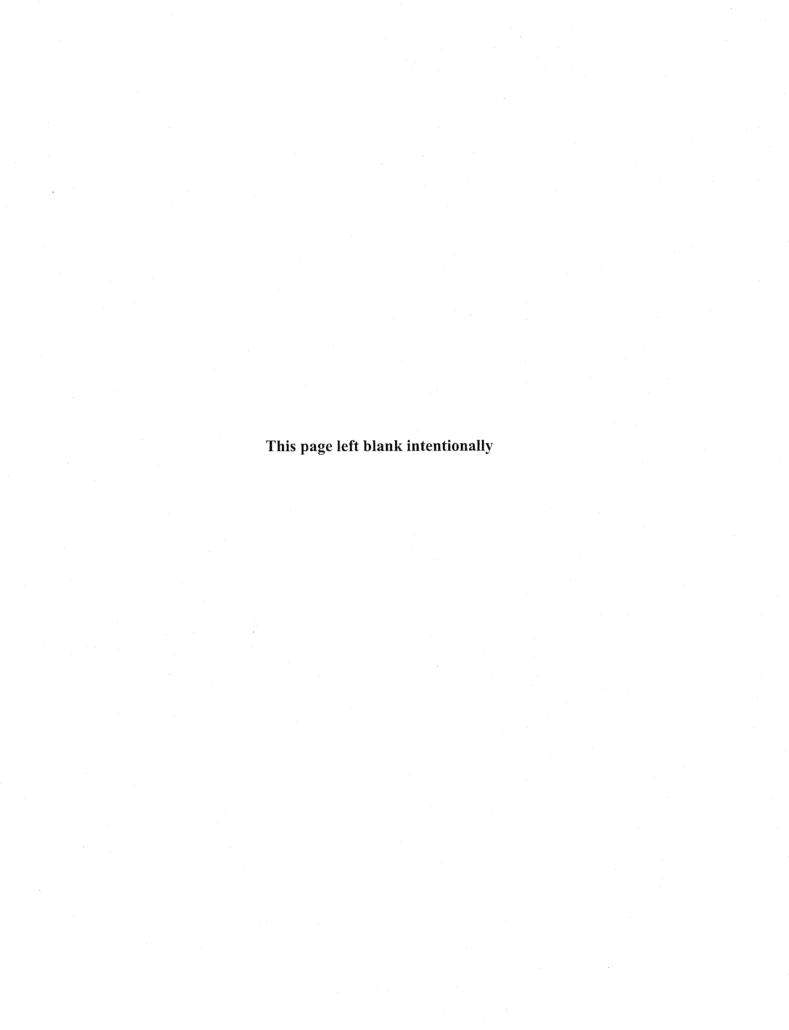
The manufacturer's expiration date is used when indicated. If none is supplied, the following expiration dates are applied:

Compound	Expiration
Gases in Liquid	1 month after opening ampule
Other Volatile compounds	6 months after opening ampule
Semivolatile/Pesticides/PCBs	1 year after opening ampule

Liquid Stock Standards Prepared from Neat Materials

Compound	Expiration
Gases in Liquid	1 month
Other Volatile Compounds	6 months
Semivolatile/Pesticides/PCBs	1 year

^{*} Used for Initial Calibration only



5.0 SAMPLE HANDLING

5.1 SAMPLING MEDIA AND PRESERVATION REQUIREMENTS

General guidelines regarding sampling media, preservation, and holding time requirements are summarized in Exhibit 5.1. The laboratory first refers to project specific requirements. These requirements can be found in the individual Statement of Work (SOW) or Quality Assurance Program Plan (QAPP). If there are no project specific guidelines, the lab uses the criteria presented in Exhibit 5.1.

Disclaimer: ATL assumes no real or implied responsibility or liability for client-related field sampling and shipping activities. It is the responsibility of the individual client to ensure that referenced methodologies are followed with respect to sample collection and shipment to the laboratory. Air sampling media and equipment should only be used by experienced field engineers. It is the ultimate responsibility of the client to be knowledgeable both in sample preservation requirements as well as relevant State, Federal, and International shipping requirements. Any time a chemical substance is collected using ATL media, the client bears sole responsibility to understand and abide by the laws involving shipment of potentially hazardous substances by common carrier.

5.1.1 Sample Containers

Items provided by the laboratory include:

- Sampling media such as Tenax®, Anasorb®-747, charcoal traps, Carbon Molecular Sieve (CMS) tubes, SummaTM polished canisters, pack250s, Tedlar bags, PUF/XAD and DNPH impinger solution
- Chain-of-custody forms
- Sampling labels
- Chemical ice packs
- Shipping containers
- Custody Seals (per client request)

• Sample Acceptance Policy

Air sampling media prepared by the laboratory for field use must be certified for cleanliness. Tenax®, Anasorb®-747, charcoal traps, Carbon Molecular Sieve (CMS) tubes, PUF/XAD and DNPH impinger solutions are certified for each preparation batch. The canister cleaning process is certified on a 10% frequency basis. Individually certified canisters are also available per specific client request.

5.1.1.1 Summa™ Canisters

The Support Services Department has dedicated approximately 1400 ft² for canister cleaning and certification functions. Approximately 200 maximum canisters can be cleaned daily and up to 60 canisters individually certified daily. This area is also sufficient for storage of approximately 600 canisters and the entire in-house inventory of flow controllers (see Table 2.2).

Ten percent of all canisters that are cleaned per ATL SOP #7 are certified by GC/MS analysis for TO-14A/15 target compounds. 6.0 L and 1.0 L canisters are certified to be clean to 0.2/0.5 ppbv for the standard product TO-14A/15 target compound list (see Section 6.8 of this document).

If a canister fails certification, the cleaning process is repeated. The canister is not returned to the inventory until it has passed certification. More information on the preparation and certification of SummaTM canisters can be found in ATL SOP #7.

ATL recommends use of 100% certified canisters for projects that require Low Level or SIM TO-14A/15 analysis and PAMS analysis. Client Services Representatives document requests of this nature in the Project Profile to assure that all shipped media meet this requirement. Canisters and associated sampling train equipment intended for projects that are defined as Low Level or SIM (see

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Section 6 Table 6.8.2) are certified at or below the Limit of Quantitation. Canisters used for PAMS analysis are certified below the Limit of Quantitation. An increase in the percent of canisters certified is also recommended for projects of a very sensitive nature.

Each ATL canister is barcoded, allowing the history of the canister to be maintained through a canister tracking system. The database keeps information, such as, date shipped, client name, date received, and the analytical work order number.

5.1.1.2 Sorbent Tubes

Each batch of sorbent tubes is certified using the prescribed analytical methodology which commonly most is either SW-846 5041A/8260B or Modified EPA TO-17. One set of tubes from each preparation batch are stored at 4°C ± 2°C and then certified by GC/MS analysis. Certification is performed before the media is shipped or used to collect samples. The background level of each target VOC must be less than the project reporting limits. The client may allow an exception to this criterion for Methylene Chloride but must provide written documention to verify acceptance. The client will be notified in advance of sampling if the batch fails and a replacement shipment provided if necessary. Tube certification results are reported with the individual field sample reports. information of the preparation and certification of sorbent tube media can be found in ATL SOP #4.

5.1.1.3 Polyurethane Foam (PUF)/XAD Cartridges

PUF and PUF/XAD cartridges are batch cleaned using a large soxhlet extraction apparatus per SOP #14. The weekly cleaning capacity is 400 PUF/XAD cartdriges and approximately 150 units are stored on-site and available for shipping at any point in time (see Table 2.2). For Method TO-13A, two PUFs from each batch are cleaned and extracted and

analyzed by GC/MS. For Methods TO-4A and TO-10A, one PUF from each batch is extracted and analyzed by GC/ECD. For Method TO-13A XAD media, 20 mls of the media is extracted and analyzed by GC/MS.

The background level of each target analyte must be less than the project reporting limits. Analysis of the extract is performed typically prior to shipping or at very least within 24 hours of shipping. The client is notified in advance of sampling if the batch fails and a replacement shipment is provided if necessary.

5.1.1.4 2,4-Dinitrophenylhydrazine (DNPH) Impinger Solution and Cartridges

The DNPH solution is prepared in bulk solution. An aliquot is removed, extracted and analyzed according to the particular method (CARB 430, Method 0011, Compendium Methods TO-5 and TO-11A). The DNPH reagent and the TO-11A media are certified as acceptable when the concentration of each analyte in the certification is below the reporting limit. In the case that contamination is present above the reporting limit; the source must be identified and eliminated prior to shipping of the reagent and the TO-11A media. If the contamination can not be eliminated, the presence of any aldehyde necessitates a call to the client. The data user, particularly in the case of source testing, may tolerate concentrations of target aldehydes near the reporting limit. Certification is completed before solution is shipped to the field. Results are shipped with the media, faxed to the site, or kept on file in the laboratory.

More information of the preparation and certification of DNPH media can be found in ATL SOP #62.

5.2 SAMPLE COLLECTION PROCEDURES – FIELD GUIDELINES

5.2.1 Information for Canister Sampling

Air Toxics Ltd. provides a technical "how to" booklet on canister sampling. The booklet includes the following information:

AIR TOXICS'

GUIDE TO AIR SAMPLING AND ANALYSIS

Fifth Edition

1.0 Introduction

1.1 Whole Air Sampling of VOCs

2.0 Canisters and Associated Media

- 2.1 Introduction to Canisters
- 2.2 Associated Canister Hardware

3.0 Sampling with Cansiters

- 3.1 Grab Samples
- 3.2 Integrated Samples

4.0 Sampling with Tedlar Bags

- 4.1 Introduction to Tedlar Bags
- 4.2 Tedlar Bag Sampling

5.0 Special Consideration Sampling

- 5.1 Special Sampling Configurations
- 5.2 Considerations for Sampling at Altitude
- 5.3 Considerations for Soil/Landfill Gas Sampling
- 5.4 Considerations for Sample Cylinder Sampling

Tables

- 1.2 Comparison of Canisters to Tedlar Bags
- 2.2.3 Fill Times for Canisters
- 3.2.3 Flow rates For Selected Sampling Intervals
- 3.2.4 Relationship Between Final Canister Vacuum, Volume Sampled and Dilution Factor

5.2.2 Information for Sorbent Tube Sampling

A copy of field sampling guide for sorbent sampling. Contents of the guide include:

AIR TOXICS'

GUIDE TO SORBENT-BASED SAMPLING VOLATILES AND SEMI-VOLATILES

First Edition

- Introduction to Air Sampling
- Introduction to Sorbents

Advantages

Disadvantages

Calculating the sample volume

Effects of moisture on sorbent efficiency

· Sorbents for VOC's

Table 1. Sorbent Characteristics

 Method Specific Guidelines for Sample Collection VOST 0030/0031/5041A/8260B

TO-17

Common sorbent tube sampling trains Returning the sorbent tube to the laboratory What if the tubes are over-sampled?

· Sorbents for SVOC's

Table 2. SVOC Sorbent Based Methods

· Method specific guidelines for sample collection

TO-4A

TO-10A

TO-13A

Modified sampling media for indoor air

Table 3. The effect of sample volume on RL's

Returning the glass cartridge to the laboratory

• What if the Tubes are Over-Sampled?

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The information provided in ATL's Sampling booklet is meant to serve only as general guidelines. In all cases, field sampling personnel are ultimately responsible for having expertise and knowledge in air sampling methodology sufficient to ensure that the defensibility of the data will not be compromised due to deficiencies in field sampling, handling or transportation.

5.3 SAMPLE RECEIVING

Upon arrival at the laboratory, samples are received and inspected following a written Sample Acceptance Policy. The Technical Director ensures that all samples are accepted in accordance with this Policy. The policy establishes specific guidelines for sample acceptance, which are generally accepted practices under EPA, AFCEE, USACE, Navy, and NELAP protocols. When samples do not meet the established guidelines, discrepancies are documented and the client is notified. Samples are noted in the individual work order and discrepancies noted in the Laboratory Narrative portion of the sample report. The Sample Acceptance Policy is provided to field staff with every shipment of containers or media.

5.3.1 Sample Acceptance Policy

Samples received by Air Toxics Ltd. must be relinquished following standard EPA approved guidelines. These include full and complete Chain-of-Custody documentation indicating:

- Unique sample name
- Location, date, and time of collection
- Collector's name
- Preservation type (if applicable)
- Matrix
- Any special remarks

The chain-of-custody form must be filled out in ink and indicate proper preservation and use of sample container specified by the method. Each sample should be labeled with unique, durable, and indelible identification and must be of adequate volume for the tests requested. Never affix a label directly on a SummaTM canister. A tag is attached to each canister for this purpose.

Proper, full, and complete inspection and documentation will be performed upon laboratory receipt in the following areas:

- evidence of container's physical damage
- status of the container's custody seal
- presence or absence of a chain-of-custody form
- incomplete or incorrect chain-of-custody form
- number of samples
- name of each sample
- sample collection date/time
- sample location
- name of the collector
- preservation type (if applicable)
- sample type (canister, XAD, DNPH etc.)
- sample tag information complete
- temperature (when applicable)
- pressure (canisters)
- presence of unlabelled samples
- presence of mis-labelled samples
- presence of unused media
- method required trip blanks, field blanks, equipment blanks, field duplicates, or field spikes

Any sample discrepancies against the above criteria are documented on the Sample Discrepancy Form (Exhibit 5.3), and communicated to the client via Login Fax within 1 day of sample receipt. The client is contacted by the project manager for discrepancies of a more serious nature, e.g.,

- Chain-of-Custody Record was not received with sample(s).
- Analysis method(s) is(are) not specified.
- Sample(s) received out of holding time.

- Sample container (Tube/VOA vial) was received broken.
- Container for VOA analysis received with headspace.
- Tedlar Bag received leaking.
- Tedlar Bag received flat.
- Tedlar bag / canister received emitting a strong odor (sample cannot be analyzed).

Documentation of client notification is included on the form along with any instructions from the client on how to proceed. Project managers complete this section and return the form to the receiving group to complete the login process. The form is archived in the Work Order folder. Whenever there is any uncertainty of how the laboratory is to proceed or when the desired method is unclear, the receiving staff places the Login process ON HOLD and delivers the Work Order file to a project manager for follow-up. The project manager contacts the client to clarify the situation. Phone calls between the project manager and the client are documented in the Client Services Software. The phone contact and client instructions to resolve the issue are logged into the database and a hardcopy report is placed in the Work Order folder. The folder is then returned to the Receiving team to complete the Login process. Air bills, packing lists, chain-of-custody records, and any other documentation that may accompany the samples are placed in the work order folder.

Laboratory malfunctions occurring during/after sample receipt are documented via the laboratory Corrective Action system. Examples of receiving problems, which would necessitate a Corrective Action Request, include:

- Hold time expired due to laboratory error.
- Canister sample pressurized with wrong type of gas.
- Sample placed On Hold was released in error.

- Sample logged in for incorrect analysis method.
- DANGER tag was not affixed to an odiferous canister sample before sending to the lab.
- Canister was released and cleaned before second analysis method was run.
- Receiving did not affix the multiple analysis tag.
- Canister valve was left open following pressurization. Sample vented to ambient.

5.3.2 The Login E-Mail/Fax

When Login is completed, an email is sent to the client to confirm receipt of samples. If no email address exists a fax is sent for the identical purpose. The Login Email/Fax has five parts:

- Page 1 Cover page with discrepancies noted
- Page 2 Log-in summary (sample names etc.)
- Page 3 Reporting template showing referenced method, target compound list, and reporting limits
- Part 4 Copy of COC
- Part 5 Media outstanding (if relevant)

Discrepancies are noted on the cover page using a template of pre-approved statements. The QA Dept. is responsible for maintaining the approved template. Receiving staff electronically copy relevant statements from the template and onto the Email/FAX cover page. Typical statements include:

- NELAC Chapter 5 specifies that a legal Chain of Custody must accompany samples when they arrive at the laboratory. In this case a chain of custody was not received with the samples. The discrepancy was noted in the Login email.
- The COC form was not completed properly. Please note for future reference

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that the COC must be signed and dated in order to properly relinquish samples.

- Samples were received past the recommended hold time of hours/days. ATL will proceed with the analysis unless otherwise notified.
- The samples do not have any documentation regarding the date of collection. Unless otherwise notified, the COC relinquish date will be used.
- The Tedlar bag for sample ____ was received flat and could not be analyzed.
- All samples were received at or near ambient pressure yet flow controllers were used. ATL will proceed with the analysis unless otherwise notified.
- Samples were not received at the recommended temperature. ATL will proceed with the analysis unless otherwise notified.

5.3.3 The Work Order Folder

A folder is created during the Login process to hold all relevant documents. The folder is labeled with the unique Work Order number, client name and analysis. One folder for each desired analysis is created so that laboratory analyses can be efficiently handled as separate processes. The folder contains the following receiving documents:

- Login summary sheet with individual field sample names, dates of collection and project reference
- Specific method cited, and a copy of the reporting target compound template for review
- Original COC record, airbill, and any other packing documents

- Original copy of the Sample Discrepancy Report
- Original copy of any CAR Forms
- Copy of the Project Management Project Profile with associated special analysis and reporting requirements
- Copy of any approved Project Requirement tables generated after the bid has been won

The folder is passed to the analytical teams after Login, and follows the same process stream as the samples. All original documents generated during the processing of the samples are filed in this folder. The unique Work Order file makes archival and retrieval of evidentiary and custodial documents easier. The majority of analytical documentation is archived electronically. Documentation that remains in hard copy form includes:

- COC
- Data Review Checklist
- Sample Discrepancy Reports
- Corrective Action Requests
- Scan Packets (run logs, spectral defenses, manual integrations etc.)
- Phone contacts and emails
- Bid Ships/Canister Certifications
- Fed-Ex/UPS air bill/freight bill
- GC/FID screening results

Alternatively, the Work Order folder is placed in a bar coded storage box for long-term storage. Work Order inventory of each box is taken prior to offsite storage and maintained along with the bar code address. A private storage company archives the boxes by barcode and provides one-day retrieval service upon request. Alternatively, work order folders may be scanned onto CD-ROM Media and stored on-site.

5.4 SAMPLE TRACKING PROCEDURES

After samples have been inspected, they are given a unique tracking number and logged into an electronic sample receiving database. The tracking number consists of the year and month plus a sequential Work Order number. As an example, the first set of samples received in July, 2004 would have the format:

0407001

If this set of samples consisted of eight individual samples, then each sample is identified by a consecutive postscript such as:

0407001-01A through 08A

If more than one analysis is requested for the samples, an alphabetic designation is given to each analysis sample set:

0407001A-01A TO-15 0407001B-01A TO-3

Laboratory assigned duplicates are designated using a double postscript such as:

0407001-01AA

A more detailed discussion of the sample receiving function is given in ATL SOP #50. The laboratory processes thousands of samples each month divided into hundreds of individual work orders. An efficient userfriendly database is critical in keeping track of each individual sample, monitoring hold times, monitoring due dates, and scheduling analyses. In addition, most air projects have specific target compound lists, reporting limit requirements, quality assurance requirements, analysis requirements, and data submission requirements. Relevant project information is immediately available as each processing step occurs. The ultimate goal of the ATL sampleprocessing system is to deliver what the customer wants the first time. Report re-issues and sample re-analyses are monitored and kept to a minimum. In order to meet the quality objective (customer satisfaction), every team

member has access to information describing what the customer has requested.

The sample tracking database consists of a variable number of data fields sufficient to store project and sample batch information. The users can then query any field in the database. Each department creates work lists from the database and inputs relevant information (e.g., completion dates, etc.) throughout the day.

The database resides on a secured network server equipped with a daily-automated back up system. Multiple PCs are available to each team in their respective work areas. Access privileges are defined and maintained by the IT team. The database is designed such that work order status can be determined at any point in time. The 'status' field is updated each time the work progresses to a new stage in its processing. Status data include:

- Client Services
- Extractions
- Log-in
- Lab Bins
- Individual Instrument Assignment
- Data Review
- OA
- FAX
- EDD Generation
- Final Report
- Financial Hold
- · Filed

Complete documentation of sample processing is maintained in the database. Each team completes relevant portions of the database as work is finished. Selected information includes:

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SAMPLE TRACKING FIELDS

Work Order number Client Services contact Date received Client name Project name Project ID number #Samples Date sampled #Lab dups **#Sample holds** Container type **Expiration date** Method specific analysis code Date promised Rush turn 24 hour clock Screen done Date receiving done Receiving analyst initials Log-in date Log-in analyst initials Date analysis done Date reported Bench analyst initials Date of final report CVP due date **Date CVP completed** Date CVP shipped CVP analyst initials EDD due date EDD completed date Date EDD shipped EDD analyst initials Reissue due date Reissue reason Time Due

The electronic database is used to document and ensure that analytical hold times, reporting requirements, and project specific QC requirements are met. The database is used by the Client Service Representatives to provide project specific activity reports and status of incomplete work. Users may query the database and easily produce a printed report.

The sample database is the key to efficient information transfer and, as such, is a critical tool to meet the quality objective.

5.5 INTERNAL SAMPLE CUSTODY AND STORAGE PROCEDURES

The chain-of-custody for samples is documented from time of receipt until time of disposal. Internal sample chain-of-custody documentation consists of:

- Storage area logbooks
- Instrument run logs
- Raw analytical data for samples, calibrations and QC checks

The samples are stored in the custody cage, in a secure refrigerator, or in the event of late delivery in the receiving section until the next morning. The receiving staff logs the samples into the Internal Sample or Extractable Sample Tracking Logbook in the storage area.

Samples are tracked in/out of the limited access area by initials, date, and time. All staff members have access to the storage areas and all members are trained on proper custody documentation in the logs. Logbook protocol training is mandatory for all staff. The training and documentation of training is handled by the QA team. The QA team checks the Logbook Review Checksheet monthly to ensure that the analysts have reviewed their logbooks on a timely basis.

5.6 SAMPLE DISPOSAL

Samples are released for disposal upon satisfactory completion of analysis unless prior contractual arrangements have been made. The release of samples is documented in the Internal Sample Tracking Log via a "Released" stamp that includes the date and initials of the person who releases the sample for disposal. Samples are released by the Laboratory Director, Technical Director, Analytical Department Managers and Team

Leaders, Laboratory Scientists or qualified Analysts.

Sample disposal varies based on the sampling media. Whole air samples are vented through a charcoal scrubber, while liquid (i.e., solvent and water) samples are disposed of according to the procedures noted in ATL's Chemical Hygiene Plan.

5.7 SUBCONTRACTING

Air Toxics Limited subcontracts samples on an infrequent basis. Subcontracting is generally performed for contractual reasons in fields of testing which the laboratory does not perform. In the event that subcontracting is necessary, the project manager selects a suitable subcontract laboratory that meets the project specified certification criteria. Work that falls under the scope of NELAC accreditation shall be placed with a laboratory accredited under NELAP for the tests to be performed or with a laboratory that meets applicable statutory and regulatory requirements for performing the tests and submitting the results of tests performed. The laboratory performing the subcontracted work shall be indicated in the final report and non-NELAP accredited work shall be clearly identified. If the project has no criteria, then the project manager selects a subcontract laboratory based on state certification in the desired field of testing. The laboratory shall advise the client of the arrangement in writing and, when possible, gain the approval of the client, preferably in writing.

Exhibit 5.1.

Requirements for Containers, Preservation Techniques, and Holding Times

Method	Parameter	Type	Container I	Preservation	Extraction A	
			***************************************		Holding Time	Holding Time
VOST	VOCs	GC/MS	Sorbent Tube	4°C	NA	14 days
5041A/82601						
TO-3 &	BTEX/TPH	GC/FID/PID	Summa Canister		NA	30 days
CARB 410A			Tedlar Bag	NA	NA	3 days
TO-4A & TO-10A	Pesticides PCBs	GC/ECD	PUF	4°C	7 days	40 days
TO-5 & CARB 430	Aldehydes & Ketones	HPLC/UV	DNPH Impinger	4°C	7 days	30 days
NIOSH 2546		GC/FID	XAD-7 Tube	4°C	2 days	30 days
TO-11A	Aldehydes & Ketones	HPLC/UV	Sep-PAK	4°C	14 days	30 days
TO-12	NMOC	GC/FID	Summa Canister	NA	NA	30 days
			Tedlar Bag	NA	NA	3 days
TO-13A/ 8270	PAHs/ Semivolatiles	GC/MS	XAD/PUF	4°C	7 days	40 days
TO-14A/15	VOCs	GC/MS	Summa Canister	NA	NA	30 days
			Tedlar Bag	NA	NA	3 days
TO-17	VOCs	GC/MS	Sorbent Tube	4°C .	NA	30 days
ASTM	Fixed	GC/TCD/FID	Summa Canister	NA	NA	30 days
D1946	Gases CH ₄ , C ₂ +		Tedlar Bag	NA	NA	3 days
ASTM	Fixed &	GC/TCD/FID	Summa Canister	NA	NA	30 days
D1945	Natural Gases		Tedlar Bag	NA	NA	3 days
ASTM D5504	Sulfur Gases	GC/SCD	Tedlar Bag	NA	NA	24 hours
Method 0011	Aldehydes & Ketones	HPLC/UV	DNPH Impinger	4°C	7 days	30 days
EPA Meth. 25C/3C	Fixed Gases & NMOC	GC/TCD/FID	Summa Canister (6 liter)	NA	NA	30 days
Headspace	Dissolved	GC/FID	40 ml VOA	4°C &	NA	14 days
Analysis	Gases		vials	1:1 HCl, pH<		
	CO_2	GC/TCD		4°C & No HCl	NA	7 days
PM10/TSP	Particulate Matter	Analytical Balance	Quartz Filter	59°F – 86°F RH< 45%	NA	14 days

Exhibit 5.2 Chain-of-Custody

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TeleId Sample I.D. (Location) Can# Date Time Analyses Requested Notes:			Date/Time	y: (signature)	Received t		1	ned by: (signature)	Relinquist
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Exhibit 5.3 Sample Discrepancy Form

Sample Discrepancy Report

	If Section III or IV is filled out CSR must	be n	notified within 24 hrs of initiation
Initiate	ed By: Date:	Gi	Given To: ☐ File to folder
	Sections I – II/III/IV must be filled out by person		
	· · · · · · · · · · · · · · · · · · ·		
I. W	orkorder(s) affected:		***************************************
S	ample(s) affected:		. Additional and the second se
	mple Receipt Discrepancies (Document on Cover Pactes of Lab Narrative)	ge of	of Sample Receipt Confirmation and in Receiving
-	COC improperly relinquished / received.		Flow controller used - canister samples received a
	COC was not filled out in ink.		ambient or under pressure.
	Sample tags / labels do not match the COC.		No brass cap on canister (do not narrate).
	Samples received at wrong temperature (# 4±2 °C); ice / blue ice (circle one) was present. A temp. blank		 VOA vial for RSK-175 analysis received with headspace bubble <5mm (do not narrate).
	was / was not present (circle one).		Other (describe below).
	Sample container (Tube/VOA vial) was received broken, <i>however</i> sample was intact.		
	ibe the Discrepancy:		
	mple Receipt Discrepancies requiring CSR notification infirmation and in Receiving Notes of Lab Narrative)	n (do	locument on Cover Page of Sample Receipt
	COC was not received with samples.		Tedlar Bag received leaking / flat (circle one).
	Analysis method(s) is not specified /		Sample can / cannot (circle one) be analyzed.
_	incorrectly specified (circle one) on the COC.		Canister leaked to ambient during pressurization.
	Number of samples on the COC does not match the number of samples that were received.		Tedlar bag / canister received emitting a strong od sample can / cannot (circle one) be analyzed.
	Samples were received expired. Sampling date / time is not documented for <u>some</u> /		Canister sample received at >15"Hg (<u>not</u> identified as a Trip/Field Blank).
	any samples (circle one).		Trip Blank received at low vacuum (< 25"Hg).
	Samples received at wrong temperature;		Tedlar Bag for Sulfur analysis has metal fitting.
	no coolant present / coolant melted (circle one). Sample container (Tube/VOA vial/DNPH Bottle, etc.)		Incorrect sampling media / container for analysis requested.
	was received broken / leaking (circle one). VOA vial for RSK-175 analysis received with		Custody Seal on the outside of the container was broken / improperly placed (circle one).
	headspace bubble >5mm. Samples for RSK-175 CO ₂ analysis received		Other (describe below).
	preserved with HCl.		
Descri	be the Discrepancy:		
Initials	:: Date:		

Revised 04/02/04

Air Toxics Ltd.

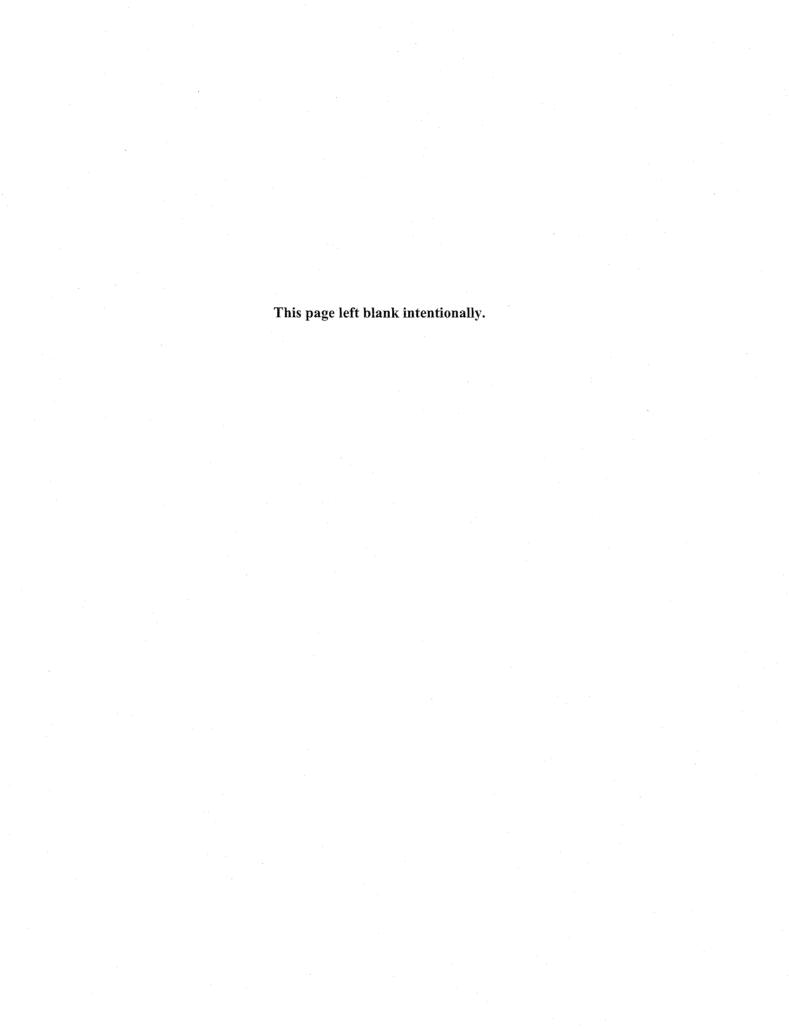
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Exhibit 5.3 Sample Discrepancy Form (Continued)

IV. <u>Lat</u>	Discrepancies requiring	Team Leader/CSR notific	ation (document in Analytical Notes of Lab Narrative)
	Tediar Bag found to be lea analysis; sample can / car			Sulfur samples received with insufficient time to analyze prior to expiration.
.—	analyzed.			VOST tube saturated; bag dilution necessary.
	Tedlar Bag found to be fla Canister found to be leaking	•		Sample loss due to instrument malfunction / broken glassware.
		•	П	Other (describe below).
	be analyzed.	v volume, sample cannot	u	Other (describe below).
Descri				
Analys	st Initials:	Date:		
Team	Leader Initials:	Date:		
V. Clie	nt Services Notification:			
	Narrate the discrepancy; i documentation on Cover Rarrative.	t is not necessary to call the Page of Sample Receipt Co	e client. onfirmati	See attached email from CSR requesting on and in Receiving Notes/Analytical Notes of Lab
	CSR notified:	Notified by:		Date:
	Narrate the discrepancy; i Confirmation and in Recei	t is not necessary to call the iving Notes/Analytical Notes	e client. s of Lab	Document on Cover Page of Sample Receipt Narrative.
	CSR Initials:	Date:		
Client	Notification:			
***************************************	notified:	Notified by:		Date:
	See attached client contact	ct / email, or comments bel	ow:	
Lab No	otification:			
Persor	notified:	Notified by:		Date:

Air Toxics Ltd.

Revised: 04/02/04



6.0 ANALYTICAL METHODS AND PROCEDURES

This section contains subsections for each analytical procedure. Each subsection contains the following information:

- A brief method description
- Laboratory variances to Compendium and SW-846 methodologies
- A table of LOQs and QC acceptance criteria
- A table of calibration procedures and QC procedures

This Quality Manual references methods in a general manner. The specific revisions used by the laboratory can be found in the method-specific SOPs.

6.1 VOST SW-846 5041A/8260B

This method involves GC/MS full scan analysis of volatile organic compounds in air

samples collected on Tenax/Charcoal (VOST) cartridges. Samples are collected using SW-846 Method 0030/0031 Volatile Organic Sampling Train (VOST) protocols. VOST cartridges are thermally The desorbed by heating and purging with Ultra High Purity Helium. The resulting gaseous effluent is then bubbled through 5 ml of organic free reagent grade water and trapped on the sorbent trap of the purge and trap system. The trap is then thermally desorbed for GC/MS analysis. For condensate analysis, a 5 ml aliquot of condensate sample is placed directly in the sparge vessel of the purge and trap (P&T) system and analyzed in a similar manner.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-1.1. Summary of Method Modifications

Requirement	EPA Method 5041A/8260B	Air Toxics Ltd. Modifications
Method Blank	Cartridges from the same media batches as the samples.	Media batch is certified prior to use in the field. Method Blank is from a different batch unless requested by the client.
Connection between thermal desorption apparatus & purge vessel.	PTFE 1/16" Teflon tubing.	Heated, 1/16" silica lined stainless steel tubing.
Calibration Criteria for non-CCCs.	RSD ≤ 15 % for all non-CCCs.	RSD ≤ 30 % for Acetone, Bromoform, Vinyl Acetate, Bromomethane, Chloromethane, 1,1,2,2- Tetracholoroethane, & 1,2,3- Trichloropropane.

Table 6-1.2. SW-846 Modified Method 5041A Standard Analyte List

Table 6-1.2. SW-846 Modified		Legidiual		ice Criteria
Analytes	RL	ICAL	LCS	
	(ng)	(%RSD)	(% R)	CCV
1,1,1-Trichloroethane	10	15	70 – 130	-
1,1,1,2-Tetrachloroethane	10	15	70 - 130	
1,1,2,2-Tetrachloroethane – SPCC	10	30	70 – 130	RF > 0.30
1,1,2-Trichloroethane	10	15	70 – 130	
1,1-Dichloroethane – SPCC	10	15	70 - 130	RF > 0.10
1,1-Dichloroethene – CCC	10	30	70 – 130	$\%D \le 25\%$ VOST tubes; $\le 20\%$ condensates
1,1-Dichloropropene	10	15	70 – 130	
1,2,3-Trichlorobenzene	50	15	70 – 130	. •
1,2,3-Trichloropropane	10	30	70 – 130	-
1,2,4-Trichlorobenzene	50	15	70 – 130	
1,2,4-Trimethylbenzene	10	15	70 – 130	_
1,2-Dibromo-3-chloropropane	50	15	70 – 130	-
1,2-Dichlorobenzene	10	15	70 – 130	-
1,2-Dichloroethane	10	15	70 – 130	-
1,2-Dichloropropane – CCC	10	30	70 – 130	$\%D \le 25\%$ VOST tubes; $\le 20\%$ condensates
1,3,5-Trimethylbenzene	10	15	70 – 130	_
1,3-Butadiene ¹	50	30	50 – 150	
1,3-Dichlorobenzene	10	15	70 - 130	-
1,3-Dichloropropane	10	15	70 – 130	-
1,4-Dichlorobenzene	10	15	70 - 130	-
2,2-Dichloropropane	10	15	70 - 130	-
2-Butanone ²	50	30	50 – 150	-
2-Chloropropane	10	15	70 - 130	-
2-Chlorotoluene	10	15	70 - 130	-
2-Hexanone ²	50	30	50 – 150	-
3-Chloropropene	.10	15	70 - 130	-
4-Chlorotoluene	10	15	70 - 130	<u>-</u>
4-Methyl-2-pentanone ²	50	30	50 - 150	-
Acetone ²	50	30	50 – 150	
Acrylonitrile	10	15	70 - 130	-
Benzene	10	15	70 - 130	_
Bromobenzene	10	15	70 – 130	м.
Bromochloromethane	10	15	70 – 130	
Bromodichloromethane	10	15	70 – 130	-
Bromoform – SPCC	10	30	70 – 130	RF > 0.10
Bromomethane ²	10	30	50 - 150	er-
Butylbenzene	10	15	70 130	
Carbon Disulfide	10	15	70 – 130	

	ът	Acceptance Criteria			
Analytes	RL	ICAL	LCS		
	(ng)	(%RSD)	(% R)	CCV	
Carbon Tetrachloride	10	15	70 - 130	-	
Chlorobenzene – SPCC	10	15	70 - 130	RF > 0.30	
Chloroethane	10	.15	50 - 150	-	
Chloroform – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates	
Chloromethane – SPCC	10	30	50 – 150	RF > 0.10	
cis-1,2-Dichloroethene	10	15	70 – 130	-	
cis-1,3-Dichloropropene	10	15	70 – 130	-	
cis-1,4-Dichloro-2-butene	50	15	70 - 130	-	
Cumene	10	15	70 – 130	. =	
Dibromochloromethane	10	15	70 - 130	_	
Dibromomethane	10	15	70 – 130	_	
Ethylbenzene – CCC	10	30	70 – 130	%D \leq 25% VOST tubes; \leq 20% condensates	
Ethylene Dibromide	10	15	70 – 130	-	
Freon 11	10	15	70 – 130		
Freon 12	10	15	50 – 150	÷ .	
Freon 113	10	15	70 – 130	<u>+</u>	
Hexachlorobutadiene	50	15	70 – 130	<u>.</u>	
Hexane	10	15	70 – 130	-	
Iodomethane	50	15	70 – 130		
Methylene Chloride	10	15	70 – 130		
Methyl t-butyl ether (MTBE)	10	30	70 - 130	-	
Naphthalene	50	15	70 - 130	-	
m,p-Xylene	10	15	70 - 130		
o-Xylene	10	15	70 - 130	-	
p-Cymene	10	15	70 - 130	·	
Propylbenzene	10	15	70 - 130	-	
sec-Butylbenzene	10	15	70 - 130		
Styrene	10	15	70 - 130	-	
tert-Butylbenzene	10	15	70 - 130		
Tetrachloroethene	10	15	70 – 130	<u> </u>	
Toluene – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates	
trans-1,2-Dichloroethene	10	15	70 – 130	-	
trans-1,3-Dichloropropene	10	15	70 - 130	-	
trans-1,4-Dichloro-2-butene	50	15	70 - 130		
Trichloroethene	10	15	70 - 130	· -	
Vinyl Acetate ^{1,2}	50	30	50 – 150	_	
Vinyl Bromide ¹ (Bromoethene)	50	30	50 - 150	<u>.</u>	
Vinyl Chloride – CCC	10	30	50 – 150	$\%D \le 25\%$ VOST tubes; $\le 20\%$ condensates	

¹ Independent source verification check not available for these compounds.

Table 6-1.3. Matrix Spike/Matrix Spike Duplicate

Analyte	%R
1,1-Dichloroethene	60 – 140
Benzene	60 – 140
Trichloroethene	60 – 140
Toluene	60 – 140
Chlorobenzene	60 - 140

Table 6-1.4. Internal Standards

Analyte	CCV IS (%R)	Sample IS (%)R
1,4-Dichlorobenzene-d ₄	50 – 200	60 - 140
Chlorobenzene-d ₅	50 – 200	60 - 140
Fluorobenzene	50 – 200	60 – 140

Table 6-1.5. Surrogates

Analyte	%R
1,2-Dichloroethane-d ₄	70 – 130
4-Bromofluorobenzene	70 – 130
Dibromofluoromethane	70 – 130
Toluene-d ₈	70 – 130

Table 6-1.6. Summary of Calibration and QC Procedures for SW-846 Modified Method 5041A

Note: These criteria are used specifically for the standard list of analytes listed in Table 6-1.2.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at the start of every 12-hour clock.	Method 5041A tuning criteria.	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis.	SPCC criteria in Table 6-1.2, CCC and non-CCC compound criteria in Table 6.1.2.	Correct problem then repeat initial calibration.
Laboratory Control Sample (LCS)	Once per initial calibration, and with each analytical batch (maximum of 20 samples).	See Table 6-1.2.	Investigate the problem and if warranted, analyze a new analytical curve for the out-of-limits compound. (except for compounds noted in Table 6-1.2.)

² Due to nature of these compounds, recoveries outside of noted limits do not result in recalibration

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration	At the start of every	For SPCCs: see "CCV criteria" column	Investigate and correct the problem, up to and including
Verification	shift immediately after the BFB tune	For CCCs: %D ≤ 25%	recalibration if necessary.
(CCV)	check.	for VOST tubes and \leq 20% for condensates.	
Internal Standards	As each standard, blank, and sample	For CCVs: area counts 50% - 200%, RT w/in 30	CCV: inspect and correct system prior to sample analysis.
(IS)	is being loaded.	sec of mid-point in ICAL.	For blanks: inspect the system and re-analyze the blank.
		For blanks, samples	For condensates: re-analyze; if
		and non-CCV QC Checks: area counts 60	out again, flag data. For VOST: flag the data,
		- 140%, RT w/in 20 sec. of RT in CCV.	evaluate system and correct problem before proceeding.
Surrogates	With all samples and QC.	See Table 6-1.5.	Same as for Internal Standards.
Laboratory	Immediately after	Results less than	Inspect the system and re-analyze
Blanks	the calibration	laboratory reporting limit	the blank.
	standard or after		·
	samples with high concentrations		
	$(\geq 5000 \text{ ng}).$		
(MS/MSD)	Once/batch of	See Table 6-1.3.	Q-flag and narrate.
	condensate samples.		

6.2 TO-3 - BTEX AND TPH

This method involves GC analysis of whole air samples collected in Summa™ canisters or Tedlar bags. Samples are analyzed for Benzene, Toluene, Ethylbenzene, Xylenes, and Total Petroleum Hydrocarbons (TPH) using EPA Method TO-3 protocols. Samples are analyzed using a Photo Ionization Detector (PID) and a Flame Ionization Detector (FID). Depending on the

client's request, TPH is analyzed and referenced to either gasoline or jet fuel.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, RL, QC criteria, and QC summary can be found in the following tables.

Table 6-2.1. Summary of Method Modifications

Requirement	EPA Method TO-3	Air Toxics Ltd. Modifications
Preparation of Standards	Levels achieved through dilution of gas mixture.	Levels achieved through loading various volumes of the gas mixture.
Initial Calibration Calculation	4-point calibration using a linear regression model.	5-point calibration using average Response Factor.
Initial Calibration Frequency	Weekly.	When daily calibration standard recovery is outside 75-125%, or upon significant changes to the procedure or instrumentation.
Daily Calibration Standard Frequency	Prior to sample analysis and every 4-6 hrs.	Prior to sample analysis and after the analytical batch ≤ 20 samples.
Minimum Detection Limit (MDL)	Calculated using the equation DL = A+3.3S, where A is intercept of calibration line and S is the standard deviation of at least 3 reps of low level standard.	40 CFR Pt. 136 App. B.
Moisture Control	Nafion system.	Sorbent system.

Table 6-2.2. Method TO-3 Standard Analyte List

	RL	Acceptance Criteria			
Analyte	(ppmv)	ICAL (%RSD)	LCS & CCV (%R)	Precision (%RPD)	
Benzene	0.001	≤ 30	± 25	≤ 25	
Toluene	0.001	≤ 30	± 25	≤ 25	
Ethyl Benzene	0.001	≤ 30	± 25	≤ 25	
Total Xylenes	0.001	≤ 30	± 25	≤ 25	
TPH* (Gasoline Range)	0.025	≤ 30	± 25	≤ 25	
TPH** (JP 4 Range)	0.025	≤ 30	± 25	≤ 25	

^{*} TPH referenced to Gasoline (MW = 100)

^{**} TPH referenced to JP 4 (MW = 156)

Table 6-2.3. Surrogate

Surrogate	PID Accuracy (%R)	FID Accuracy (%R)
Fluorobenzene	75-125%	75-150%

Table 6-2.4. Summary of Calibration and OC Procedures for Method TO-3 (BTEX & TPH)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five Point Calibration (ICAL)	Prior to sample Analysis.	%RSD ≤ 30.	Repeat the calibration.
Laboratory Control Sample (LCS)	With each initial calibration, and with each analytical batch.	±25% of the expected value.	Check the system and re-analyze the standard. Re-prepare the standard or re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis.	±25% of the expected value.	For initial CCV: Check the system and re-analyze the standard. Recalibrate the instrument if the criteria cannot be met.
Mid/End Check	At the end of the analytical batch, not to exceed 20 samples.	±25% of the expected value.	Check system and re-analyze the standard. If the 2nd analysis fails, identify and correct the problem, then re-analyze all samples since the last acceptable CCV.
Laboratory Blank	In between analysis of standards and project samples.	Results less than the laboratory Reporting Limit.	Inspect the system and re-analyze the Laboratory Blank.
Surrogate Spikes	As each standard, blank, and sample is being loaded.	75-125% recovery on the PID, 75-150% on the FID.	Low surrogate recovery results in reanalysis (at a higher dilution if high levels of moisture are present). If recovery is out and still low, report the analysis with the better recovery and flag. Because of TPH interference, high surrogate recoveries do not result in re-analysis. Data is flagged to note high recovery.
Laboratory Duplicates	10% of the samples.	RPD \leq 25% for detections $>$ 5 X's RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found on the system, narrate the data.

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6.3 TO-4A/TO-10A – PESTICIDES AND PCBS

This method involves GC analysis of Pesticides and Aroclor Polychlorinated Biphenyls (PCBs) in ambient air samples collected on polyurethane foam (PUF) cartridges.

Adsorbent PUF cartridges are cleaned using solvents and vacuum dried. Cartridges are sent to the field wrapped tightly in aluminum foil to prevent degradation by ultraviolet (UV) light. The PUF cartridges are batch certified for cleanliness prior to shipping. In addition, the laboratory analyzes one clean PUF cartridge for each extraction set to serve as a Laboratory Blank.

A high volume sampler is used for TO-4A and a low volume sampler is used for method TO-10A. A glass fiber filter may also be included in the sampling scheme in order to collect particulate bound matter.

The filters and cartridges are prepared for analysis by either Soxhlet or Pressurized Fluid Extraction (PFE) by EPA Method 3545A. The extract is concentrated, exchanged into Hexane and concentrated again to final volume. Analysis is performed using a GC/ECD (Electron Capture Detector).

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-3.1. Summary of Method Modifications for TO-4A/TO-10A

Requirement	EPA Method TO-4A/TO-10A	Air Toxics Ltd. Modifications
Extraction Solvent	10 % (5 % TO-10A) Diethyl Ether in Hexane.	DCM, exchanging to Hexane during the concentration step.
Reagent Blank	Set up extraction system without filter/PUF; reflux with solvent.	No Reagent Blank is extracted. Reagent lots are certified as acceptable prior to use.
Media certification (TO-10A only)	< 0.01 µg for single peak analytes, < 0.1 µg for PCBs.	< Reporting Limit for all analytes.
Frequency of Continuing Calibration Verification	Every 10 samples.	Every 20 samples with internal standard.
PCB Quantitation	Requires a minimum of 5 peaks.	Use 4 peaks for quantitation.

Table 6-3.2. Methods TO-4A/TO-10A Pesticides and PCBs Reporting and QC Limits

Table 0-3.2. Wethous		Acceptance Criteria				
Analyte	RL (μg)	ICAL (%RSD)	ISCV (%R)	CCV (%D)	LCS ² (%R)	Precision (%RPD)
4,4'-DDD	0.10	< 20	± 15	± 15		≤ 25%
4,4'-DDE	0.10	< 20	± 15	± 15		≤ 25%
4,4'-DDT	0.10	< 20	± 15	± 15	65-125	≤ 25%
4,4'-Methoxychlor	1.0	< 20	± 15	± 15		≤ 25%
Aldrin	0.10	< 20	± 15	± 15	65-125	≤ 25%
alpha-BHC	0.10	< 20	± 15	± 15		≤ 25%
alpha-Chlordane	0.10	< 20	± 15	± 15		≤ 25%
Aroclor 1016/1242	1.0	< 20	± 15	± 15	65-125	≤ 25%
Aroclor 1221 [®]	1.0	< 20	± 15	± 15		≤ 25%
Aroclor 1232 [©]	1.0	< 20	± 15	± 15		≤ 25%
Aroclor 1248 [©]	1.0	< 20	± 15	± 15		≤ 25%
Aroclor 1254 [®]	1.0	< 20	± 15	± 15		≤.25%
Aroclor 1260	1.0	< 20	± 15	± 15	65-125	≤ 25%
beta-BHC	0.10	< 20	± 15	± 15		≤ 25%
delta-BHC	0.10	< 20	± 15	± 15		≤ 25%
Dieldrin	0.10	< 20	± 15	± 15	65-125	≤ 25%
Endosulfan I	0.10	< 20	± 15	± 15		≤ 25%
Endosulfan II	0.10	< 20	± 15	± 15		≤ 25%
Endosulfan Sulfate	0.10	< 20	± 15	± 15		≤ 25%
Endrin	0.10	< 20	± 15	± 15	65-125	≤ 25%
Endrin Aldehyde	0.10	< 20	± 15	± 15		≤ 25%
Endrin Ketone	0.10	< 20	± 15	± 15		≤ 25%
gamma-BHC (Lindane)	0.10	< 20	± 15	± 15	65-125	≤ 25%
gamma-Chlordane	0.10	< 20	± 15	± 15		≤ 25%
Heptachlor	0.10	< 20	± 15	± 15	65-125	≤ 25%
Heptachlor Epoxide	0.10	< 20	± 15	± 15		≤ 25%
Technical Chlordane ^{©®}	1.0	< 20	± 15	± 15		≤ 25%
Toxaphene [⊕]	1.0	< 20	± 15	± 15		≤ 25%

① The noted multi-component compounds use a one-point calibration.

Table 6-3.3. Surrogates 3

Surrogate	%R
TCMX	$60 - 120^{\circ}$
DCB	60 − 120 [©]

² Recovery limits are derived from Compendium Method TO-10A January, 1999.

③ Recovery limits are for extracted samples only. Non-extracted samples use limits of 85 – 115 %R.

Not routinely reported, available at client request

Table 6-3.4. Summary of Calibration and OC Procedures for Methods TO-4A/TO-10A

	Minimum		corrective			
QC Check	Frequency	Acceptance Criteria	Action			
Five Point ICAL*	Prior to sample analysis.	%RSD ≤ 20 for each compound or average %RSD ≤ 20.	Use linear regression per SW-846 or re-calibrate.			
Independent Source Calibration Verification (ISCV)	Calibration.		Investigate the source of discrepancy, including re-preparation and reanalysis of standard. Re-calibrate if needed.			
Breakdown Check	Daily, CCV Pesticides only.	Degradation $\leq 15\%$.	Perform maintenance. Repeat breakdown check.			
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, every 20 samples, and at the end of the analysis sequence.		Analyze new ICAL and/or prepare fresh standards. If the standard analyzed is high and associated samples are ND, "Q" flag the high recoveries. If the standard analyzed is low, re-analyze all samples.			
Laboratory Control Spike (LCS)	Extracted with each set of up to 20 samples.	As mentioned in Table 6-3.2.	Analyze another aliquot. If it still fails, "Q" flag the compounds outside the control limits.			
Surrogates	All samples, QC, and blanks prior to extraction.	As mentioned in Table 6-3.3	Analyze another aliquot, if it still fails, "Q" flag the compounds outside the control limits.			
Internal Standard	With all analyses.	CCV 50-200% compared to midpoint of ICAL; Samples 50-200% compared to first CCV of the daily analytical batch.	Analyze another 100 µL aliquot. If a CCV fails, correct problem before proceeding. If a sample fails, analyze a second time. If it still fails, dilute the sample until IS meet the criteria. Narrate the matrix interference.			
Laboratory Blanks	With each set of up to 20 samples extracted.	Results less than the Laboratory reporting limit.	Analyze another aliquot. If it still fails, "B" flag the compounds that do not meet the acceptance criteria.			
Laboratory Duplicates	10% of the samples.	RPD \leq 25% for detections $>$ 5 X's RL.	Analyze sample a 3 rd time. If criteria are still not met, narrate the data.			
Second- Column Confirmation	100% for all positive results, for both Pesticide /PCB analyses.	Same as for initial or primary column analysis.	Same as for initial or primary column analysis.			

^{*} A single point calibration is performed for Technical Chlordane, Toxaphene, and certain Aroclors.

6.4 TO-5/CARB 430, METHOD 0011, AND TO-11A- ALDEHYDES AND KETONES

These methods involve High-Pressure Liquid Chromatography (HPLC) analysis of Aldehydes and Ketones in stationary and ambient air samples. The sampling media consist either of various-sized impingers containing 2,4-Dinitrophenylhydrazine (DNPH) reagent, or a Sep-PAK (silica) cartridge coated in-situ with a solution of DNPH. Aldehydes and Ketones are readily converted to a stable Hydrazone derivative. The impinger contents are extracted with a

70:30 Methylene Chloride/Hexane solution or Methylene Chloride only, concentrated, solvent exchanged and analyzed on the HPLC. The Sep-PAK cartridges are eluted with Acetonitrile using gravity feed technique. Analysis is performed by reverse phase HPLC with UV detection at 360nm.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-4.1. Summary of TO-5/CARB 430 Method Modifications

Requirement	TO-5/CARB 430	Air Toxics Ltd. Modifications		
Initial Calibration (ICAL)	TO-5: Linear regression, $R \ge 0.999$.	Average Response Factor (RF), composite % RSD ≤ 10. Linear regression is performed when requested.		
Sample Quantitation	Use daily RF.	Use ICAL RF.		
Calibration Standard Precision	% RSD ±10%.	Recovery of all Continuing Cal. standards must be 90% - 110%.		
Retention Time (RT) Precision	%RPD < 2% for daily calibration standards.	RT windows determined by bracketing standards.		
Limit of Detection	CARB 430: The "limit of detection" is defined as the upper bound of the 95% confidence interval for the analysis of at least 4 reagent blanks.	Detection Limit is based on the current MDL study which is calculated from a minimum of 7 extracted spikes following 40 CFR Part 136 Appendix B.		
Field Blank Subtraction	CARB 430: Subtract the average of the field blanks from the samples.	The samples and Field Blanks are not blank subtracted.		
Laboratory Control Spike (LCS)	CARB 430: If the LCS is out it must be re-extracted until it is in or recalibrate.	The LCS is only extracted once with out of control recoveries flagged.		
UV Absorption Detector	TO-5: Operate at 370 nm.	Operate at 360 nm.		
Mobile Phase	TO-5: Methanol/Water.	Acetonitrile/Water.		

Table 6-4.2. Summary of Method TO-11A Modifications

Requirement	TO-11A	Air Toxics Ltd. Modifications		
Initial Calibration Curve	Multi-point using linear	Multi-point using average		
(ICAL)	regression performed	Response Factor; re-calibration if		
	every 6 months.	daily cal. fails, major maintenance		
		or column change. Linear		
		regression is performed when		
		requested.		
ICAL Criteria	R2 for curve > 0.999	%RSD ≤ 10% unless Linear		
	*	regression is requested.		
Blank Subtraction	Average blank	One Lab Blank is analyzed per		
	concentrations calculated.	batch; no blank subtraction		
	Blank value subtracted	performed on samples.		
	from sample result.			

Table 6-4.3. Methods TO-5/CARB 430, TO-11A, and Method 0011 Standard Analyte List

	TO-5/	,	Acceptance Criteria			
Analyte	Method TO-11A 0011/ RL CARB430 (μg) RL (μg)		ICAL (%RSD)	ISCV (%R)	CCV (%R)	
Formaldehyde	0.5	0.05	≤ 10	± 15	± 10	
Acetaldehyde	0.5	0.10	≤ 10	± 15	± 10	
Acrolein ^a	0.5	0.25 °	≤ 10	± 15	± 10	
Acetone*	NA	0.25	≤ 10	± 15	± 10	
Propanal	0.5	0.25	≤ 10	± 15	± 10	
Crotonaldehyde*	0.5	0.25	≤ 10	± 15	± 10	
n- Butyraldehyde ^b	0.5	0.25	≤ 10	± 15	± 10	
Isopentanal*	0.5	0.25	≤ 10	± 15	± 10	
Pentanal	0.5	0.25	≤ 10	± 15	± 10	
m,p-Tolualdehyde*	0.5	0.25	≤ 10	± 15	± 10	
o-Tolualdehyde*	0.5	0.25	≤ 1.0	± 15	± 10	
Hexanal	0.5	0.25	≤ 10	± 15	± 10	
Dimethylbenzaldehye **	0.5	0.25	≤ 10	± 15	± 10	

Because its derivative is not stable, when the target analyte list includes Acrolein, the sample will need to be extracted in field. A special order should be placed with the laboratory during the project set up stage.

Methyl Ethyl Ketone, Iso-Butyraldehyde and the n-Butyraldehydes co-elute(report as n-Butyraldehyde)

Not recommended for TO-11A

^{*} Not included in the extracted LCS compound list for methods CARB 430/TO-5 and method 0011.

^{**} Special request compound

Table 6-4.4. Summary of Calibration and QC Procedures for Methods TO-5/CARB 430, TO-11A, and Method 0011

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five Point Initial Calibration Curve (ICAL)	Analyzed in triplicate prior to sample analysis	%RSD ≤ 10.	Repeat calibration.
Instrument LCS	With each ICAL	%R = 85-115%.	Check the system and re- analyze the standard. Re- calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, after a maximum of every 10 injections, and at the end of the analytical batch.	Within \pm 10% of the expected value.	Check the system and reanalyze the standard. If the criteria cannot be met, recalibrate the instrument. If the standard is biased low, reanalyze all samples since last acceptable CCV. If biased high and samples are "ND", reanalysis is not required. Q-flag high recoveries.
Instrument (Solvent) Blank Analysis	Following analysis of Standards.	Results less than the laboratory RL.	Inspect the system and Re-analyze the blank.
Laboratory Duplicates	10% of samples.	RPD ≤ 25% for detections >5 Xs RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

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6.5 NIOSH 2546 – CRESOLS AND PHENOLS

This method involves GC/FID (Flame Ionization Detector) analysis for Phenol and Methylphenols (Cresols) in ambient air samples. The sampling media consists of solid two section sorbent tubes containing 100mg/50mg XAD-7. A sample size of 5 to 24 Liters is collected. The sorbent media is

sonicated and desorbed separately in 2.0 mL Methanol. A desorption efficiency study is necessary for each XAD-7 lot.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-5.1. Summary of Method Modifications

Requirement	NIOSH 2546	Air Toxics Ltd. Modifications		
Desorption Study	Conducted once per year.	Conducted once per sorbent lot.		
Sample Quantitation	Corrected for desorption efficiency.	Not corrected for desorption efficiency.		
Calibration Standard	Calibrate daily with at least six working standards.	Initial calibration with at least six points (%RSD \leq 20%). Continuing Calibration Verification at the start and at the end of each analytical batch (%D \leq 25%).		

Table 6-5.2. NIOSH 2546 Standard Analyte List

	RL (µg)	Low Point of the Curve (μg)	Acceptance Criteria			
Analyte			ICAL (% RSD)	LCS (% R)	CCV (% R)	Precision (% RPD)
2-Methylphenol (o-Cresol)	1.0	1.0	≤ 20	75 - 125	75 - 125	< 25%
m,p-Cresol	1.0	1.0	≤ 20	75 - 125	75 - 125	< 25%
Phenol	1.0	1.0	≤ 20	75 - 125	75 - 125	< 25%

Table 6-5.3. Summary of Calibration and QC Procedures for NIOSH 2546

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Six-point ICAL	Prior to sample analysis.	% RSD ≤ 20.	Correct problem then repeat Initial Calibration.
Independent Source Check Standard (LCS)	All analytes – once per Initial Calibration.	75 - 125 % of the expected value.	Investigate the problem and if warranted, analyze a new analytical curve for the out-of-limits compound.
Continuing Calibration Verification (CCV)	Daily, prior to sample analysis, every 20 samples, after last sample of the day, as an End Check.	75 - 125 % of the expected value.	Check the system and re-analyze the standard. If the criteria cannot be met, re-calibrate the instrument.
Laboratory Blanks	With each extraction batch.	Results less than laboratory RL.	Inspect the system and re-analyze the blank. If contamination still present, "B" flag associated sample detections.
Laboratory Control Spikes (LCS)	With each extraction batch.	% Recovery between 75-125 %.	"Q" flag the compounds outside the acceptance criteria.
Sample Duplicates	10 % of the samples.	RPD \leq 25 % for detections $>$ 5X the RL.	Re-analyze the sample a 3 rd time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.
Desorption Study	For every sorbent tube lot.	Reported upon client request.	NA

6.6 TO-12 - NMOC

This method involves GC analysis of whole air samples collected in SummaTM canisters or Tedlar bags. Samples are analyzed for Non-Methane Organic Compounds (NMOC) using EPA Method TO-12 protocols. After concentration on a sorbent bed, samples are analyzed using a Flame

Ionization Detector (FID). This method is used when speciation is not required.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Reporting Limits, QC criteria, and QC summary can be found in the following tables.

Table 6-6.1. Summary of Method Modifications

Requirement	EPA Method TO-12	Air Toxics Ltd. Modifications
Reporting Limit	0.02 ppmc.	0.010 ppmv.
Reported Units	Ppmc.	Ppmv, or if ref to CH ₄ , multiply by 7. Units in ppmvc.
Initial Calibration	Five levels – each level three runs with %RSD < 3%; Linearity criterion not specified.	Minimum of three single levels; %RSD ≤ 30%.
Sample Analysis Frequency	Duplicate analysis with RPD <5%, report average result.	Single analysis. Duplicate 10% of samples with RPD \leq 25% for detections \geq 5 X's the RL.
Sample Hold Time	None specified.	Canister 30 days, Tedlar bags 3 days.
Column	GC column not used.	GC column used for analysis.

Table 6-6.2. Method TO – 12 Standard Analyte List

Analyte	RL	ICAL	CCV	LCS	Duplicates
	(ppmv)	(%RSD)	%D	%R	%RPD
TNMOC ref. to Heptane	0.010	≤ 30	± 25	75-125	≤ 25

Heptane MW = 100

Table 6-6.3. Summary of Calibration and QC Procedures for Method TO-12

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Curve (ICAL)	Prior to sample analysis.	$\%$ RSD \leq 30.	Repeat the calibration.
Laboratory Control Sample (LCS)	With each initial calibration and analytical batch.	75-125% of the expected value.	Check the system and re-analyze the Standard. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis and after every 20 samples or at the end of the analytical sequence.	% Difference ± 25.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met. Re-analyze all samples since the last acceptable CCV.
Laboratory Blank	In between analysis of standards and project samples.	Results less than laboratory reporting limit.	Repeat the Laboratory Blank. If the reanalysis of the Lab Blank contains above but at less than 5 X the reporting limit, sample analysis may proceed and the associated sample results will be reported with a B flag.
Sample Duplicates	10% of the samples.	≤ 25% for detections > 5X's the RL.	Re-analyze the sample for a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

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6.7 TO-13A AND 8270C – SEMIVOLATILE COMPOUNDS

This method involves GC/MS full scan or SIM mode analysis of semi-volatile organic compounds in ambient air samples collected on PUF/XAD2 cartridges. In relation to the prescribed media, sampling and collection efficiency for compounds not listed in TO-13A has not been evaluated. Samples are prepared by either soxhlet or Pressurized Fluid Extraction (PFE) by EPA Method 3545A and analyzed for Polynuclear Aromatic Hydrocarbons (PAHs) using a quadrupole GC/MS in full scan or SIM mode by TO-13A protocol. In addition, the target compound list is often extended to

include analysis of Method 8270 semi-volatile compounds. Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Air Toxics Ltd. also performs semi-volatile analysis by SW-846 Method 8270C. The extraction process of MM5 trains follows SW846 Method 3542, and the QC criteria differ from Method TO-13A analysis. The QC criteria and QC summary tables for Method 8270C analysis are in the section following the TO-13A tables.

Table 6-7.1. Summary of Method Modifications for TO-13A

Requirements	EPA Method TO-13A	Air Toxics Ltd. Modifications
Extraction Solvent	10% ether in hexane for PUF; DCM for XAD sorbent. Final extract in hexane.	DCM for PUF/XAD cartridge and XAD sorbent. Final extract in DCM.
Glassware Cleaning	Muffle furnace is utilized.	Solvent cleaning procedure is used.
Extraction Technique	Soxhlet extraction.	Soxhlet extraction or pressurized fluid extraction (PFE).
Reporting List	19 PAHs.	See Tables 6-7.2 & 6-7.3.
Calibration range:	0.1-2.5 μg/mL in Hexane	1.0-160 μg/mL in Methylene chloride for quad or 0.1-40 μg/mL for SIM.
Surrogate	Field surrogates: Fluoranthene-d10 and Benzo(a)pyrene-d12.	Field surrogates: provided upon request.
Solvent Process Blank	One each analytical batch.	Not performed: each solvent lot is certified.
Method Blank	< MDL.	<reporting limit.<="" td=""></reporting>

Table 6-7.2. Modified Method TO-13A

Analyte	SIM RL (µg)	RL (μg)	Minimum ICAL RRF	ICAL (%RSD)	ISCV (%R)	CCV (%R)	Precision (%RPD)
2-Chloronaphthalene*	0.1	1.0	NA	≤30	± 50	± 30	≤ 25%
2-Methylnaphthalene*	0.1	1.0	NA	≤30	± 50	± 30	≤ 25%
Acenaphthylene	0.1	1.0	1.3	≤30	± 50	± 30	≤ 25%
Acenaphthene	0.1	1.0	0.8	≤ 30	± 50	± 30	≤ 25%
Anthracene	0.1	1.0	0.7	≤30	± 50	± 30	≤ 25%
Benzo(a)anthracene	0.1	1.0	0.8	≤30	± 50	± 30	≤ 25%
Benzo(e)pyrene*	0.1	1.0	NA	≤30	± 50	± 30	≤ 25%
Benzo(a)pyrene	0.1	1.0	0.7	≤ 30	± 50	± 30	≤ 25%
Benzo(b)fluoranthene	0.1	1.0	0.7	≤30	± 50	± 30	≤ 25%
Benzo(g,h,i)perylene	0.1	1.0	0.5	≤ 30	± 50	± 30	≤ 25%,
Benzo(k)fluoranthene	0.1	1.0	0.7	≤30	± 50	± 30	≤ 25%
Chrysene	0.1	1.0	0.7	≤30	± 50	± 30	≤ 25%
Dibenz(a,h)anthracene	0.1	1.0	0.4	≤30	± 50	± 30	≤ 25%
Fluoranthene	0.1	1.0	0.6	≤30	± 50	± 30	≤ 25%
Fluorene	0.1	1.0	0.9	≤30	± 50	± 30	≤ 25%
Indeno(1,2,3-c,d)pyrene	0.1	1.0	0.5	≤30	± 50	± 30	≤ 25%
Naphthalene	0.1	1.0	0.7	≤30	± 50	± 30	≤ 25%
Phenanthrene	0.1	1.0	0.7	≤30	± 50	± 30	≤ 25%
Pyrene	0.1	1.0	0.6	< 30	± 50	± 30	≤ 25%

^{*} Not included in the TO-13A method.

The following two compounds can be analyzed upon client's request.

Analyte	SIM RL (µg)	RL (µg)	Minimum ICAL RRF	ICAL (%RSD)	ISCV (%R)	CCV (%R)	Precision (%RPD)
Perylene	NA	1.0	0.5	≤ 30	± 50	± 30	≤ 25%
Coronene	NA	1.0	0.7	≤ 30	± 50	± 30.	≤ 25%

Table 6-7.3. Modified Method TO-13A-Extended

Analyte	Minimum ICAL RRF	RL (µg)	ICAL (1) (%RSD)	ISCV (%R)	Precision %RPD
1,2,4-Trichlorobenzene	NA	1.0	≤ 30	± 50	≤ 25%
1,2-Dichlorobenzene	NA	1.0	≤ 30	± 50	≤ 25%
1,3-Dichlorobenzene	NA	1.0	≤ 30	± 50	≤ 25%
1,4-Dichlorobenzene - CCC	NA	1.0	≤ 30	± 50	≤ 25%
2,4,5-Trichlorophenol	NA	5.0	≤ 30	± 50	≤ 25%
2,4,6-Trichlorophenol - CCC	NA	5.0	≤ 30	± 50	≤ 25%

Analyte	Minimum	RL (μg)	ICAL (1)	ISCV (%R)	Precision %RPD
2.4 Dishlararkard CCC	RRF	5.0	< 30	± 50	Z 250/
2,4-Dichlorophenol - CCC	NA NA				≤ 25% ≤ 25%
2,4-Dimethylphenol	NA NA	5.0	<u>≤30</u>	± 50	≤ 25%
2,4-Dinitrophenol - SPCC	0.05	20	≤ 30	± 50	≤ 25%
2,4-Dinitrotoluene	NA	5.0	<u>≤30</u>	± 50	≤ 25%
2,6-Dinitrotoluene	NA	5.0	<u>≤30</u>	± 50	≤ 25%
2-Chloronapthalene	NA	1.0	≤ 30	± 50	≤ 25%
2-Chlorophenol	NA	5.0	<u>≤ 30</u>	± 50	≤ 25%
2-Methylnapthalene	NA	1.0	≤ 30	± 50	≤ 25%
2-Methylphenol	NA	5.0	≤ 30	± 50	≤ 25%
2-Nitroaniline	NA	10	≤30	± 50	≤ 25%
2-Nitrophenol – CCC	NA	5.0	≤ 30	± 50	≤ 25%
3,3-Dichlorobenzidine	NA	20	<u>≤30</u>	± 50	≤ 25%
3-Nitroaniline	NA	10	≤ 30	± 50	≤ 25%
4,6-Dinitro-2-methylphenol	NA	10	≤ 30	± 50	≤ 25%
4-Bromophenyl-phenyl ether	NA	1.0	≤ 30	± 50	≤ 25%
4-Chloro-3-methylphenol - CCC	NA	5.0	≤ 30	± 50	≤ 25%
4-Chloroaniline	NA	10	≤ 30	± 50	≤ 25%
4-Chlorophenyl-phenyl ether	NA	1.0	≤ 30	± 50	≤ 25%
4-Methylphenol	NA	5.0	≤ 30	± 50	≤ 25%
4-Nitroaniline	NA	10	≤ 30	± 50	≤ 25%
4-Nitrophenol – SPCC	0.05	20	≤ 30	± 50	≤ 25%
Acenaphthylene	1.3	1.0	≤ 30 ·	± 50	≤ 25%
Acenaphthene – CCC	0.8	1.0	≤ 30	± 50	≤ 25%
Anthracene	0.7	1.0	≤ 30	± 50	≤ 25%
Benzo(a)anthracene	NA	1.0	≤ 30·	± 50	< 25%
Benzo(a)pyrene - CCC	0.7	1.0	< 30	± 50	≤ 25%
Benzo(e)pyrene	0.5	1.0	< 30	± 50	≤ 25%
Benzo(b)fluoranthene	0.7	1.0	<u>≤</u> 30	± 50	≤ 25%
Benzo(g,h,i)perylene	NA	1.0	<u>≤</u> 30	± 50	<u>≤ 25%</u>
Benzo(k)fluoranthene	NA	1.0	<u>≤</u> 30	± 50	< 25%
Benzoic Acid	NA	30	< 30	± 50	< 25%
Bis(2-Chloroethoxy) Methane	NA	1.0	<u>≤</u> 30	± 50	< 25%
Bis(2-Chloroispropyl) Ether	NA	1.0	<u>≤30</u>	± 50	< 25%
Bis(2-Chlroethyl) Ether	NA	1.0	< 30	± 50	< 25%
Bis(2-Ethylhexyl)phthalate	NA	5.0	< 30	± 50	<u>≤ 25%</u>
Butylbenzylphthalate	NA	5.0	< 30	± 50	$\frac{-25\%}{\leq 25\%}$
Chrysene	0.7	1.0	< 30	± 50	< 25%
di-n-Butylphthalate	NA	5.0	< 30	± 50	< 25%
di-n-Octylphthalate - CCC	NA NA	5.0	< 30	± 50	≤ 25% ≤ 25%
Dibenz(a,h)anthracene	0.4	1.0	< 30	± 50	$\leq 25\%$
Dibenzofuran	NA	1.0	< 30	± 50	$\leq 25\%$
Diethylphthalate	NA NA	5.0	$\frac{30}{30}$	± 50 ± 50	$\frac{\leq 25\%}{\leq 25\%}$
Dimethylphthalate	NA NA	5.0	$\frac{\leq 30}{\leq 30}$	± 50 ± 50	$\frac{25\%}{25\%}$

Analyte	Minimum ICAL RRF	RL (µg)	ICAL (1) (%RSD)	ISCV (%R)	Precision %RPD
Fluoranthene – CCC	0.6	1.0	≤ 30	± 50	≤ 25%
Fluorene	0.9	1.0	≤ 30	± 50	≤ 25%
Hexachlorobenzene	NA	1.0	≤ 30	± 50	≤ 25%
Hexachlorobutadiene - CCC	NA	1.0	≤ 30	± 50	≤ 25%
Hexachlorocylcopentadiene- SPCC	0.05	20	≤ 30	± 50	≤ 25%
Hexachloroethane	NA	1.0	≤ 30	± 50	≤ 25%
Indeno(1,2,3-c,d)pyrene	0.5	1.0	≤ 30	± 50	≤ 25%
Isophorone	NA	1.0	≤ 30	± 50	≤ 25%
n-Nitroso-di-n-propylamine– SPCC	0.05	1.0	≤30	± 50	≤ 25%
n-Nitrosodiphenylamine – CCC	NA	10	≤ 30	± 50	≤ 25%
Naphthalene	0.7	1.0	≤ 30	± 50	≤ 25%
Nitrobenzene	NA	1.0	<u>≤</u> 30	± 50	≤ 25%
Pentachlorophenol – CCC	NA	20	≤ 30	± 50	≤ 25%
Phenanthrene	0.7	1.0	≤ 30	± 50	≤ 25%
Phenol – CCC	NA	5.0	≤ 30	± 50	≤ 25%
Pyrene	0.6	1.0	≤ 30	± 50	≤ 25%

⁽¹⁾ With 10% exception not to exceed 40%

Table 6-7.4. Surrogates (Full Scan)

Analyte	(%R)
2,4,6-Tribromophenol	50 – 150
2-Fluorophenol	50 – 150
Nitrobenzene-d ₅	50 – 150
Phenol-d ₅	50 – 150
Fluorene-d10	60 – 120
Pyrene-d10	60 - 120

Table 6-7.5. Internal Standards

Lable o 7.5. Internal St	enam as
Analyte	(%)
Acenaphthene-d ₁₀	50 – 200
Chrysene-d ₁₂	50 – 200
1,4-Dichlorobenzene-d ₄	50 - 200
Naphthalene-d ₈	50 - 200
Perylene-d ₁₂	50 - 200
Phenanthrene-d ₁₀	50 - 200

Table 6-7.6. TO-13A-Surrogates (Standard and SIM)

Analyte	Accuracy (% R)*
Fluorene-d ₁₀	60 - 120
Pyrene-d ₁₀	60 - 120

Table 6-7.7. Extracted Laboratory Control Spikes for Modified TO-13A-Extended

Analyte	(%R)
1,2,4-Trichlorobenzene***	50 – 150
1,4-Dichlorobenzene***	50 – 150
2,4-Dinitrotoluene***	50 – 150
2-Chlorophenol***	50 – 150
4-Chloro-3-methylphenol***	50 – 150

4-Nitrophenol***	50 – 150
Acenaphthene*	60 – 120
N-Nitroso-di-n-propylamine***	50 – 150
Pentachlorophenol**	22 – 109
Phenol***	50 – 150
Pyrene*	60 – 120

- * The LCS and Surrogate limits are derived from Compendium Method TO-13A Sections 13.3.7.4 and 13.4.6.3 January, 1999. These limits only apply to samples that are extracted by Air Toxics Ltd. When sample extracts are sent to Air Toxics Ltd., limits of 50 150 % are applied.
- ** Pentachlorophenol is not included in Compendium Method TO-13A and has been shown to be erratically recovered from XAD media therefore historical Control Limits are used. Limits are updated periodically as needed.
- *** Compounds outside of the TO-13A method

Table 6-7.8. Extracted Laboratory Control Samples for TO-13A (PAHs) in Full Scan and SIM

Analyte	(%R)
Napthalene	60 – 120
Acenapthylene	60 - 120
Acenaphtene	60 – 120
Flourene	60 – 120
Phenanthrene	60 - 120
Anthracene	60 – 120
Fluoranthene	60 – 120
Pyrene	60 – 120
Benzo (a) anthracene	60 – 120
Chrysene	60 – 120
Benzo (b) flouranthene	60 – 120
Benzo (k) flouranthene	60 – 120
Benzo (a) pyrene	60 - 120
Indeno (1,2,3-cd) pyrene	60 – 120
Dibenzo (a,h) anthracene	60 – 120
Benoz (g,,h,i) perylene	60 – 120

Table 6-7.9. Summary of Calibration and QC Procedures for EPA Method TO-13A

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and		Correct problem then repeat
B	at start of every 12 hrs.	for semivolatiles	tune.
		analysis. DDT%	
. '		Breakdown< 20%	·
Initial 5-Point	Prior to sample	ICAL criteria in tables	Correct problem then repeat
Calibration	analysis.	6-7.2 and 6-7.3	initial calibration.
ICAL LCS	All analytes – Once per	All target compound	Determine the source of
	initial calibration.	recoveries must be	discrepancy between standards.
		between 50 – 150%.	Re-calibrate if needed.
Continuing	At the start of every	PAHs list: meet min.	Investigate and correct the
Calibration		RRF requirement	problem, up to and including
Verification	the DFTPP tune check.	PAHs list/ short list	re-calibration if necessary.
(CCV)		$\%D \le 30\%$	High bias associated with non-
		Semivol full list:	detects in samples will not
		SPCCs: RF ≥ 0.050	result in re-analysis.
·		$\%D \le 30\%$ with 10%	,
	·	exception not to	
		exceed 40%. Flag all	
		results outside of	
		compliance with the	
		exception of high bias	. *
		associated with non-	
		detects.	
Internal	As each standard,	For CCV: Area count	For CCVs: Investigate and
Standards (IS)	blank, and sample is	within 50 to 200% of	correct the problem before
	being aliquoted.	the mid point of ICAL.	proceeding with sample
			analysis. If interferences are
		For blanks, samples	present, a secondary ion may
		and non-CCV QC	be selected.
		Checks: retention	For blanks: inspect the system
		times within ± 0.33	and re-analyze the blank.
		minutes (20 seconds) and area counts within	For samples and non-CCV QC: unless there is obvious matrix
		50 to 200% of the	effect, re-analyze the samples
		CCV.	and dilute the sample until the
		CC V.	IS meet the criteria, narrate the
			data to indicate interference.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogates	With all samples and	See Table 6-7.4.	A new aliquot of the extract is
	blanks prior to		analyzed. If Surrogate
	extraction.		recoveries are out-of-control a
			second time, data is flagged
			and narrated. Re-analysis is
			not necessary for obvious
			matrix effects (data is flagged
			for out-of-control surrogate
			recoveries). Air samples cannot
			be re-extracted.
Extracted	With each set of up to	See LCS Criteria in	Re-aliquot and re-analyze the
LCS	20 extracted samples.	tables 6-7.7 and 6-7.8.	extract. If within limits, report
	,		the re-analysis. Otherwise,
			narrate.
Laboratory	With each set of up to	Results less than	Flag the data.
Blank	20 extracted samples.	laboratory reporting	·
		limit.	
Solvent Blanks	When samples that are	All target compounds	Flag the data.
	extracted together are	below the reporting	
	analyzed on different	limit.	
	analytical shifts.		
Laboratory	10% of the samples.	RPD $\leq 25\%$ for all hits	Narrate the data.
Duplicates		> 5X RLs.	

Table 6-7.10. Summary of Method Modifications for EPA Methods 3510/3542 and 8270C

Requirements	EPA Method 8270C	Air Toxics Ltd. Modifications
Linearity of ICAL	Use mean RF for non-CCC	Use mean RF for non-CCC
	compounds if %RSD≤ 15%. If	compounds when %RSD≤ 30%.
	%RSD>15%, use a) linear	·
	regression equation that does	
	not pass through the origin. R	
	>/= 0.99, or b) non-linear (i.e.,	
	6 points for a quadratic model).	
RT for CCV	Within +/- 30 seconds of the	Frequent column maintenance results
	mid-point standard from the	in RT shift; therefore this requirement
	initial curve.	is not practical.

Table 6-7.11. SW-846 Modified Method 8270C Standard Analyte List

	Acceptance Criteria				
Analyte	RL (μg)	ICAL (0) DCD)	ISCV	CCV [®]	Precision\%
1.2.4 Trichlorohonzono	1.0	(%RSD) [©] < 15	(%R) [©]	%D < 20%	RPF ≤ 25%
1,2,4-Trichlorobenzene		 	<u>+ 50</u>	$\frac{76D \le 20\%}{\%D < 20\%}$	
1,2-Dichlorobenzene	1.0	≤ 15	<u>+ 50</u>		
1,3-Dichlorobenzene	1.0	≤ 15	<u>+ 50</u>	$\%D \le 20\%$	
1,4-Dichlorobenzene - CCC	1.0	<u>≤30</u>	<u>+ 50</u>	$\frac{\%D \le 20\%}{200\%}$	
2,4,5-Trichlorophenol	5.0	≤ 15	<u>+ 50</u>	$\%D \le 20\%$	
2,4,6-Trichlorophenol - CCC	5.0	<u>≤30</u>	<u>+ 50</u>	$\%D \le 20\%$	
2,4-Dichlorophenol - CCC	5.0	<u>≤ 30</u>	± 50	$\%D \le 20\%$	
2,4-Dimethylphenol	5.0	<u>≤ 15</u>	<u>+ 50</u>	$\%D \le 20\%$	
2,4-Dinitrophenol - SPCC	20	≤ 15	± 50	RF > 0.050	≤ 25%
2,4-Dinitrotoluene	5.0	< 15	± 50	%D ≤ 20%	≤ 25%
2,6-Dinitrotoluene	5.0	< 15	± 50	%D ≤ 20%	≤ 25%
2-Chloronaphthalene	1.0	< 15	± 50	%D ≤ 20%	
2-Chlorophenol	5.0	< 15	+ 50	%D ≤ 20%	
2-Methylnaphthalene	1.0	< 15	+ 50	$\%D \le 20\%$	
2-Methylphenol	5.0	< 15	+ 50	$^{-}$ %D $\leq 20\%$	
2-Nitroaniline	10	<u>≤</u> 30	± 50	$\frac{-}{\%D \le 20\%}$	
2-Nitrophenol – CCC	5.0	<u>≤</u> 30	+ 50	$\%D \le 20\%$	
3,3-Dichlorobenzidine	20	<u>≤15</u>	± 50.	$^{-}$ %D $\leq 20\%$	
3-Nitroaniline	10	< 30	+ 50	$^{-}$ %D $\leq 20\%$	
4,6-Dinitro-2-methylphenol	10	<u>≤</u> 30	± 50	$\%D \le 20\%$	
4-Bromophenyl-phenyl ether	1.0	< 15	+ 50	$\%D \le 20\%$	
4-Chloro-3-methylphenol - CCC	5.0	< 30	+ 50	%D < 20%	≤ 25%
4-Chloroaniline	10	< 30	± 50	$\%D \le 20\%$	≤ 25%
4-Chlorophenyl-phenyl ether	1.0	<u>≤ 15</u>	± 50	%D ≤ 20%	<u>≤ 25%</u>
4-Methylphenol	5.0	< 15	± 50	%D ≤ 20%	
4-Nitroaniline	10	≤ 30	± 50	%D ≤ 20%	
4-Nitrophenol – SPCC	20	<u>≤</u> 15	<u>+</u> 50	RF > 0.050	<u>≤ 25%</u>
Acenaphthylene	1.0	< 15	+ 50	%D < 20%	< 25%
Acenaphthene – CCC	1.0	< 30	+ 50	$\%D \le 20\%$	
Anthracene	1.0	< 15	+ 50	$\%D \le 20\%$	
Benzo(a)anthracene	1.0	<u>≤15</u>	± 50	%D < 20%	
Benzo(a)pyrene - CCC	1.0	<u>− 10</u> ≤ 30	± 50	$\frac{90 - 20\%}{\text{MD}}$	
Benzo(b)fluoranthene	1.0	≤ 15	± 50	$\frac{100 - 20\%}{\text{MD} < 20\%}$	
Benzo(g,h,i)perylene	1.0	< 15	+ 50	$\frac{6D - 20\%}{D < 20\%}$	
Benzo(k)fluoranthene	1.0	< 15	+ 50	$\%D \le 20\%$	
Benzoic Acid	30	<u>≤30</u>	± 50	$\%D \le 20\%$	
Bis(2-Chloroethoxy) Methane	1.0	< 15	+ 50	$\%D \le 20\%$	
Bis(2-Chloroispropyl) Ether	1.0	< 15	+ 50	$\%D \le 20\%$	
Bis(2-Chlroethyl) Ether	1.0	<u>≤15</u>	+ 50	$\%D \le 20\%$	
Bis(2-Ethylhexyl)phthalate	5.0	< 15	± 50	%D < 20%	

	DI	Acceptance Criteria			
Analyte	RL (µg)	ICAL (%RSD) [©]	ISCV (%R) ²	CCV [®]	Precision\% RPF
Butylbenzylphthalate	5.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Chrysene	1.0	<u>≤ 15</u>	± 50	$\%D \le 20\%$	≤ 25%
di-n-Butylphthalate	5.0	≤ 15	<u>+ 50</u>	%D ≤ 20%	≤ 25%
di-n-Octylphthalate - CCC	5.0	≤ 30	<u>+</u> 50	%D ≤ 20%	≤ 25%
Dibenz(a,h)anthracene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Dibenzofuran	1.0	< 15	± 50	%D ≤ 20%	≤ 25%
Diethylphthalate	5.0	≤ 1.5	± 50	%D ≤ 20%	≤ 25%
Dimethylphthalate	5.0	<u>≤</u> 15	± 50	%D ≤ 20%	≤ 25%
Fluoranthene – CCC	1.0	< 30 ≤ 30 × 30 × 30 × 30 × 30 × 30 × 30 ×	± 50	%D ≤ 20%	≤ 25%
Fluorene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Hexachlorobenzene	1.0	≤ 15	± 50	%D ≤ 20%	≤ 25%
Hexachlorobutadiene – CCC	1.0	≤ 30	± 50	%D ≤ 20%	≤ 25%
Hexachlorocylcopentadiene –	20	<u>≤</u> 15	<u>+</u> 50	RF>	≤ 25%
SPCC Hexachloroethane	1.0	< 15	± 50	0.050 $\%D \le 20\%$	< 25%
2.74	1.0	< 15			
Indeno(1,2,3-c,d)pyrene			± 50	$\%D \le 20\%$	
Isophorone	1.0	≤ 15	<u>+ 50</u>	%D ≤ 20%	
n-Nitroso-di-n-propylamine – SPCC	1.0	≤ 15	± 50	RF > 0.050	≤ 25%
n-Nitrosodiphenylamine – CCC	10	≤ 30	± 50	%D ≤ 20%	< 25%
Naphthalene	1.0	< 15	+ 50	$\%D \le 20\%$	
Nitrobenzene	1.0	<u>≤</u> 15	<u>+</u> 50	$\%D \le 20\%$	<u>≤25%</u>
Pentachlorophenol – CCC	20	<u>≤</u> 30	± 50	%D ≤ 20%	
Phenanthrene	1.0	<u>≤ 15</u>	± 50	%D ≤ 20%	≤ 25%
Phenol – CCC	5.0	<u>≤</u> 30	<u>+</u> 50	%D ≤ 20%	≤ 25%
Pyrene	1.0	<u>≤</u> 15	<u>+</u> 50	%D ≤ 20%	≤ 25%

- \mathcal{D} Can use the mean RSD criterion of $\leq 15\%$ as noted in par. 7.5.1.2.1 of SW-846, 8000B.
- 2 No more than 10% of the target compounds are allowed to exceed the limit.
- If %D for all CCC is less than or equal to 20%, then the CCV is assumed to be valid. If the CCCs are not included in the list of analytes for a project, then all analytes must meet the 20% D.

Table 6-7.12. Surrogates

Analyte	Accuracy @ (% R)
2,4,6-Tribromophenol	10 – 123
2-Fluorobiphenyl	43 – 116
2-Fluorophenol	21 - 110
Nitrobenzene-d ₅	35 – 114
Phenol-d ₅	10 – 110
p-Terphenyl-d ₁₄	33 – 141

Table 6-7.13. Internal Standards

Analyte	Accuracy (% R)
Acenaphthene-d ₁₀	-50 to +100
Chrysene-d ₁₂	-50 to +100
1,4-Dichlorobenzene-d ₄	-50 to +100
Naphthalene-d ₈	-50 to +100
Perylene-d ₁₂	-50 to +100
Phenanthrene-d ₁₀	-50 to +100

The Surrogate limits are derived from USEPA CLP OLM 03.0 and OLM04.2. Air Toxics Ltd. receives a numerically insufficient number of liquid samples for SW 8270C analysis to allow semi-annual updating of in-house Control Limits.

Table 6-7.14. Extracted Laboratory Control Spikes Table 6-7.15. Pre-Spike Surrogates

Analyte	Accuracy <i>O</i> (% R)
1,2,4-Trichlorobenzene	39 – 98
1,4-Dichlorobenzene	36 – 97
2,4-Dinitrotoluene	24 – 96
2-Chlorophenol	27 - 123
4-Chloro-3-methylphenol	23 - 97
4-Nitrophenol	10 – 80
Acenaphthene	46 – 118
N-Nitroso-di-n-propylamine	41 – 116
Pentachlorophenol	9 – 103
Phenol	12 - 110
Pyrene	26 – 127

	Accuracy Ø (%R)
Benzo(a)Pyrene-d ₁₂	50 - 150
Fluoranthene- d ₁₀	50 – 150

- ① The LCS limits are derived from USEPA CLP OLM03.0 and OLM04.2. Air Toxics Ltd. receives a numerically insufficient number of samples for SW 8270C analysis to allow semi-annual updating of in-house Control Limits. These limits only apply to samples that are extracted by Air Toxics Ltd. When sample extracts are sent to Air Toxics Ltd., limits of 50 - 150% are applied.
- ② The pre-spike Surrogates limits are arbitrary. Air Toxics Ltd. received a numerically insufficient number of samples for SW 8270C analysis to allow semi-annual updating of inhouse control limits.

Table 6-7.16. Summary of Calibration and QC Procedures SW-846 Modified Method 8270C

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at start of every 12 hrs.	SW-846 tuning criteria for Semi- volatiles analysis.	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis.	ICAL criteria in Table 6-7.10.	Correct problem then repeat Initial Calibration.
Independent Source Calib. Ver. (ISCV)	All analytes – once per Initial Calibration.	At least 90% of the target compounds recoveries must be between 50 – 150%.	Determine the source of discrepancy between standards. Re-calibrate if needed.
Continuing Calibration Verification (CCV)	At the start of every clock, immediately after the DFTPP tune check.	SPCCs: RF≥0.050 CCCs: %D ≤20%; Non-CCC's when CCC compounds are not requested %D ≤20%.	Investigate and correct the problem, up to and including re-calibration if necessary. High bias for one or more compounds associated with non-detects in the samples will not result in re-analysis.
Internal Standards (IS)	As each standard, blank, and sample is being aliquoted.	For CCVs: area counts within -50% to +100% from the most recent ICAL. For blanks, samples and non-CCV QC Checks: Retention Times within ± 0.33 minutes (20 seconds) and area counts within -50% to +100% of the CCV.	For CCVs: Investigate and correct the problem before proceeding with sample analysis. For blanks: Inspect the system and re-analyze the blank. For samples and non-CCV QC: Reanalyze the samples. If the criteria are not met a second time, dilute sample until IS meet criteria.
Surrogates	and blanks prior to extraction.	See Table 6-7.11.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise narrate.
Extracted LCS	With each set of up to 20 extracted samples.	See LCS Criteria in Table 6-7.13.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise narrate.
Laboratory Blank	With each set of up to 20 extracted samples.	Results less than laboratory RL.	Re-aliquot and re-analyze the extract to confirm the presence of the target compound. If it doesn't confirm, investigate and correct the problem before re-analyzing all the affected samples.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Solvent Blanks	1	All target compounds below the RL.	Investigate and correct the problem before re-analyzing all the affected samples.
Laboratory Duplicates	10% of the samples.	RPD \leq 25% for all detections $>$ 5 X's RLs.	Analyze a third time. Report the closest two results and narrate and report the data if the criteria is still not met.

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6.8 TO-14A/TO-15 – VOLATILE ORGANIC COMPOUNDS

This method involves full scan GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds using EPA Method TO-14A/15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and is either concentrated using a cryogenic trap and/or concentrated using a hydrophobic multisorbent bed. The

hydrophobic multisorbent bed functions as a drying system which removes water from the sample stream prior to analysis by full scan GC/MS. For low level analysis, the sample is focused onto a cryogenic cooled column for analysis by full scan GC/MS.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-8.1. Summary of Method Modifications

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Sample Drying System	Nafion Drier.	Multisorbent.	Multisorbent.
Blank acceptance criteria	< 0.2 ppbv.	< RL.	< RL.
Blanks and standards (applies to Low Level analysis only)	Zero Air.	Zero air.	Nitrogen.
BFB absolute abundance criteria	Within 10% of that from the previous day.	Not mandated.	CCV internal standard area counts are compared to ICAL, corrective action for > 40 %D.
Daily CCV	≤ 30% D.	≤ 30% D.	≤ 30% D with two allowed out to 40% for QUAD analysis and four allowed out to 40% for Low Level analysis; flag and narrate outliers.
Initial Calibration	≤30 % RSD.	≤ 30 % RSD with 2 compounds allowed out to < 40 % RSD.	≤ 30 % RSD with 2 compounds allowed out to < 40 % for QUAD analysis and 4 compounds allowed out to < 40 % for Low Level analysis.

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Method Detection Limit	Not Specified.	Follow 40CFR Pt.136 App. B.	The MDL met all relevant requirements in Method TO-15 (statistical MDL less than the LOQ). The concentration of the spiked replicate may have exceeded 10X the calculated MDL in some cases.
Sample collection media.	Summa canister.	Summa canister.	Air Toxics Ltd. recommends use of summa canisters to insure data defensibility, but will report results from Tedlar bags at client request.

Table 6-8.2. Method TO-14A/TO-15 Analyte List (Standard Compounds)

	RL		Acceptar	ice Criteria
Analyte	(ppbv) TO- 15/LL	%RSD TO-15/LL	LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,1,2-Trichloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,1-Dichloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,1-Dichloroethene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2,4-Trichlorobenzene	2.0/0.5	30%/30%	70 - 130	≤ 25
1,2,4-Trimethylbenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2-Dibromoethane (EDB)	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2-Dichlorobenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2-Dichloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,2-Dichloropropane	0.5/0.1	30%/30%	70 - 130	≤ 25
1,3,5-Trimethylbenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,3-Dichlorobenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
1,4-Dichlorobenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
Benzene	0.5/0.1	30%/30%	70 - 130	≤ 25
Bromomethane	0.5/0.1	30%/30%	70 - 130	≤ 25
Carbon Tetrachloride	0.5/0.1	30%/30%	70 - 130	≤ 25
Chlorobenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
Chloroethane	0.5/0.1	30%/30%	70 - 130	≤ 25
Chloroform	0.5/0.1	30%/30%	70 - 130	≤ 25
Chloromethane	2.0/0.1	30%/30%	70 - 130	≤25
Chlorotoluene (Benzyl Chloride)	0.5/0.1	30%/30%	70 - 130	≤ 25
cis-1,2-Dichloroethene	0.5/0.1	30%/30%	70 - 130	≤ 25
cis-1,3-Dichloropropene	0.5/0.1	30%/30%	70 - 130	≤ 25
Dichloromethane	0.5/0.2	30%/30%	70 - 130	≤ 25
Ethylbenzene	0.5/0.1	30%/30%	70 - 130	≤ 25
Freon 11 (Trichlorofluoromethane)	0.5/0.1	30%/30%	70 - 130	≤ 25
Freon 113 (Trichlorotrifluoroethane)	0.5/0.1	30%/30%	70 - 130	≤ 25

	RL		Acceptai	nce Criteria
Analyte	(ppbv) TO- 15/LL	%RSD TO-15/LL	LCS (%R)	Precision Limits (Max. RPD)
Freon 114	0.5/0.1	30%/30%	70 - 130	≤ 25
Freon 12 (Dichlorodifluoromethane)	0.5/0.1	30%/30%	70 - 130	≤ 25
Hexachlorobutadiene	2.0/0.5	30%/30%	70 - 130	≤ 25
m,p-Xylene	0.5/0.1	30%/30%	70 - 130	≤ 25
Methyl Chloroform	0.5/0.1	30%/30%	70 - 130	≤ 25
o-Xylene	0.5/0.1	30%/30%	70 - 130	≤ 25
Styrene	0.5/0.1	30%/30%	70 - 130	≤ 25
Tetrachloroethene	0.5/0.1	30%/30%	70 - 130	≤ 25
Toluene	0.5/0.1	30%/30%	70 - 130	≤ 25
trans-1,3-Dichloropropene	0.5/0.1	30%/30%	70 - 130	≤ 25
Trichloroethene	0.5/0.1	30%/30%	70 - 130	≤ 25
Vinyl Chloride	0.5/0.1	30%/30%	70 - 130	≤ 25

Table 6-8.3. Method TO-14A/TO-15 Analyte List (Non-Standard and Polar Compounds)

	RL		Acceptar	ice Criteria
Analyte	(ppbv) TO- 15/LL	%RSD TO-15/LL	LCS (%R)	Precision Limits
1,3-Butadiene	0.5/0.5	30%/30%	60 - 140	≤ 25°
1,4-Dioxane	2.0/0.1	30%/30%	60 - 140	≤ 25 _.
2-Butanone (Methyl Ethyl Ketone)	0.5/0.1	30%/30%	60 - 140	≤ 25·
2-Hexanone	2.0/0.5	30%/30%	60 – 140	≤ 25
4-Ethyltoluene	0.5/0.1	30%/30%	60 – 140	≤ 25
4-Methyl-2-Pentanone (MIBK)	0.5/0.1	30%/30%	60 - 140	≤ 25
Acetone	2.0/0.5	30%/30%	60 – 140	≤ 25
Bromodichloromethane	0.5/0.1	30%/30%	60 - 140	≤ 25
Bromoform	0.5/0.1	30%/30%	60 - 140	≤ 25
Carbon Disulfide	0.5/0.5	30%/30%	60 - 140	≤ 25
Cyclohexane	0.5/0.1	30%/30%	60 - 140	≤ 25
Dibromochloromethane	0.5/0.1	30%/30%	60 - 140	≤ 25
Ethanol	2.0/0.5	30%/30%	60 - 140	≤ 25
Heptane	0.5/0.1	30%/30%	60 - 140	≤ 25
Hexane	0.5/0.1	30%/30%	60 - 140	≤ 25
Isopropanol	2.0/0.5	30%/30%	60 - 140	≤ 25
Methyl t-Butyl Ether (MTBE)	0.5/0.1	30%/30%	60 - 140	≤ 25
Propylene	2.0/0.5	30%/30%	60 - 140	≤ 25
Tetrahydrofuran	0.5/0.5	30%/30%	60 – 140	≤ 25
trans-1,2-Dichloroethene	0.5/0.1	30%/30%	60 – 140	≤ 25
2,2,4-Trimethylpentane	0.5/0.5	30%/30%	60 – 140	≤ 25
Cumene	0.5/0.1	30%/30%	60 - 140	≤ 25
Propylbenzene	0.5/0.1	30%/30%	60 - 140	≤ 25

RL			Acceptance Criteria	
Analyte	(ppbv) TO- 15/LL	%RSD TO-15/LL	LCS (%R)	Precision Limits
3-Chloroprene	2.0/0.5	30%/30%	60 – 140	≤ 25
TPH (Gasoline) or NMOC (Hexane/Heptane)	10	One Point Calibration	NA	≤ 25

Table 6-8.4. Internal Standards

Table 6-8.5. Surrogates

			·
Analyte	Accuracy (% R)	Analyte	Accuracy (% R)
Bromochloromethane	60 - 140	1,2-Dichloroethane-d ₄	70 – 130
1,4-Difluorobenzene	60 - 140	Toluene-d ₈	70 – 130
Chlorobenzene-d ₅	60 - 140	4-Bromofluorobenzene	70 - 130

Table 6-8.6. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW – 846 tune criteria.	Correct problem then repeat tune.
5-Point Calibration	Prior to sample analysis.	% RSD \leq 30 with two compounds allowed out to \leq 40% RSD (4 allowed out for LL).	Correct problem then repeat Initial Calibration Curve.
LCS	After each initial calibration curve, and daily, prior to sample analysis.	Recoveries for 90% of "Standard" compounds must be 70-130%; for 80% of "Non-standard" compounds, recoveries must be 60-140%. No recovery may be <50%.	Check the system and reanalyze the standard. Reprepare the standard if necessary. Re- calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	%D ≤30% for all compounds with two exceptions not to exceed 40% (four allowed out for LL). Narrate all results outside of compliance. Flag all results outside of compliance with the exception of high bias associated with non-detects.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5 point calibration curve.
Laboratory Blank	After the CCV/LCS.	Results less than the laboratory reporting limit.	Inspect the system and Re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT	For blanks: inspect the system

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
	,	in the CCV and within	sample. If the ISs are within
Decoration		±40% of the area counts of	limits in the re-analysis, report
		the daily CCV internal	the second analysis. If ISs are
		standards.	out-of-limits a second time,
			dilute the sample until ISs are
			within acceptance limits and
			narrate.
Surrogates	As each standard,	70 - 130%.	For blanks: inspect the system
	blank, and sample is		and reanalyze the blank.
	being loaded.		For samples: re-analyze the
-			sample unless obvious matrix
			interference is documented. If
<i>;</i>			the %R is within limits in the re-
			analysis, report the second
			analysis. If %R is out-of-limits
			a second time, then narrate
			results.
Laboratory	10% of the samples.	RPD ≤25% for detections	Re-analyze the sample a third
Duplicates		>5 X's the RL.	time. If the limit is exceeded
	·		again, investigate the cause and
		ĺ	bring the system back to
			working order. If no problem is
			found on the system, narrate
			results.

6.9 TO-14A/TO-15 VOLATILE ORGANIC COMPOUNDS BY SIM

This method involves Selective Ion Monitoring (SIM) GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds using EPA Method TO-14A/15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and concentrated using a cryogenic trap. The focused air sample is then flash heated through a hydrophobic drying system that

removes water from the sample stream. The sample is then focused onto a cryogenic cooled column prior to analysis by GC/MS in the (SIM) mode.

Air Toxics Ltd. performs a modified version of this method. The target analyte list and Limit of Quantitation reflect relevant risk driving compounds and are available upon request. The method modifications, QC criteria, and QC summary may be found in the following tables.

Table 6-9.1. Summary of Method Modifications

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Sampling/concentrator system	Nafion Drier.	Multi-sorbent concentrator.	Multi-sorbent concentrator
Blank acceptance criteria	< 0.2 ppbv.	< RL.	< RL.
Blank and standards	Zero air.	Zero air.	Nitrogen.
BFB absolute abundance criteria	Within 10% of that from previous day.	Not mandated.	CCV internal standard area counts are compared to ICAL, corrective action for > 40% D.
ICAL %RSD acceptance criteria	< 30% RSD.	≤ 30%, with two compounds allowed to < 40%.	Project specific; default criteria is ≤30% RSD with 10% of compounds allowed out to < 40% RSD.
Daily CCV	≤30% D.	≤30% D.	Project specific; default criteria is $\leq 30\%$ Difference with 10% of compounds allowed out up to $\leq 40\%$.; flag and narrate outliers.

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Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Method Detection Limit	Not Specified.	Follow 40CFR Pt.136 App. B.	The MDL met all relevant requirements in Method TO-15 (statistical MDL less than the LOQ). The concentration of the spiked replicate may have exceeded 10X the calculated MDL in some cases.

Table 6-9.2. Internal Standards

Table 6-9.3. Surrogates

Analyte	Accuracy (% R)	Analyte	Accuracy (% R)
Bromochloromethane	60 - 140	1,2-Dichloroethane-d ₄	70 - 130
1,4-Difluorobenzene	60 - 140	Toluene-d ₈	70 - 130
Chlorobenzene-d ₅	60 - 140	4-Bromofluorobenzene	70 – 130

Table 6-9.4 Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 by SIM

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW – 846 tune criteria.	Correct problem then repeat tune.
5-6-Point Calibration	Prior to sample analysis	≤30% for standard compounds with 10% of the compound list allowed out to ≤40% RSD.	Correct problem then repeat Initial Calibration Curve.
Laboratory Control Standard (LCS)	calibration curve, and daily prior to	Recoveries for 90% of "Standard" compounds must be $\pm 30\%$; for 80% of "Non-standard" compounds, recoveries must be $\pm 40\%$. No recovery may be $< 50\%$.	Check the system and re-analyze the standard. Re-prepare the standard if necessary. Re- calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	D with 10% of the	Perform maintenance and repeat test. If the system still fails the CCV, perform a new calibration curve.
Laboratory Blank	After the LCS.	Results less than the laboratory reporting limit.	Inspect the system and re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within ±0.33 min of the RT in the CCV and within ±40% of the area counts of the daily CCV internal standards.	For samples: re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70 - 130%.	For blanks: inspect the system and re-analyze the blank. For samples: re-analyze the sample unless obvious matrix interference is documented. If the %R is within limits in the re-analysis, report the second analysis. If %R is out-of-limits a second time, report data from first analysis and narrate.

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QC Check	Minimum	Acceptance	Corrective
	Frequency	Criteria	Action
Laboratory Duplicates	10% of the samples.	RPD \leq 25% for detections $>$ 5 X's the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

6.10 GC/MS DIRECT INJECT TO-14A/15

This method involves full scan analysis of whole air samples collected in evacuated stainless steel canisters and Tedlar bags. Samples are analyzed for volatile organic compounds using EPA Method TO-14A/15 protocols. An aliquot of the sample is withdrawn from the canister through a gas tight syringe and is injected directly onto a sorbent-based focusing interface. The

focused air sample is then flash heated through a hydrophobic drying system which removes water from the sample stream prior to analysis by full scan GC/MS.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-10.1. Summary of Method Modifications

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Sampling Drying System	Nafion Drier.	Multi-sorbent concentrator.	Multi-sorbent concentrator.
Blank acceptance criteria	< 0.2 ppbv.	< RL.	< RL.
BFB Tune absolute abundance criteria	Within 10% of that from the previous day.	Not mandated.	CCV internal standard area counts are compared to ICAL, corrective action for > 40 %D.
Initial Calibration	≤ 30 % RSD.	≤ 30 % RSD with 2 compounds allowed out to < 40 % RSD.	≤ 30 % RSD with 2 compounds allowed out to < 40 % RSD.
Daily CCV	≤ 30% D.	≤ 30% D.	≤ 30% D with two allowed out to 40%; flag and narrate outliers.
Method Detection Limit	Not Specified.	Follow 40CFR Pt.136 App. B.	The MDL met all relevant requirements in Method TO-15 (statistical MDL less than the LOQ). The concentration of the spiked replicate may have exceeded 10X the calculated MDL in some cases.

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Sample collection media	Summa canister.	Summa canister.	Air Toxics Ltd. recommends use of summa canisters to insure data defensibility, but will report results from Tedlar bags at client's request.

Table 6-10.2. Method TO-14A/TO-15 GC/MS Direct Inject Analyte List

	RL	Acceptance Criteria			
Analyte	(ppbv)	LCS <i>③</i> (%R)	Precision %RPD	ICAL Ø %RSD	CCV Ø %R
1,1,1-Trichloroethane	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
1,1-Dichloroethane	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
1,1-Dichloroethene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Benzene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Carbon Tetrachloride	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Chlorobenzene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Chloroform	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
cis-1,2-Dichloroethene	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
Ethyl Benzene	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
Freon 11 (Trichlorofluoromethane)	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Freon 113 (Trichlorotrifluoroethane)	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
Freon 114	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
Freon 12 (Dichlorodifluoromethane)	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
m,p-Xylene	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
Methylene Chloride	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
o-Xylene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Tetrachloroethene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Toluene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
trans-1,2-Dichloroethene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Trichloroethene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Vinyl Chloride	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
Chloromethane	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
2-Propanol	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
Methyl tert-Butyl Ether	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
Bromomethane	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
Chloroethane	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
1,2-Dichloroethane	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
1,2-Dichloropropane	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
1,1,2-Trichloroethane	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
1,1,2,2-Tetrachloroethane	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
1,3,5-Trimethylbenzene	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%

	RL	Acceptance Criteria			
Analyte	(ppbv)	LCS ③ (%R)	Precision %RPD	ICAL Ø %RSD	CCV Ø %R
1,2,4-Trimethylbenzene	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
1,2-Dichlorobenzene	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
1,3-Dichlorobenzene	5.0	70 - 130	≤ 25%	≤30%	≤ 30%
1,4-Dichlorobenzene	5.0	70 - 130	≤ 25%	≤ 30%	≤ 30%
1,2-Dibromoethane	5.0	70 - 130	≤ 25%	≤30%	≤30%
TPH ref. to Gasoline (MW = 100)	100	NA	≤ 25%	≤ 30%	≤ 30%
NMOC ref. to Heptane (MW = 100)	100	NA	≤ 25%	≤ 30%	≤ 30%

Table 6-10.3. Internal Standard

Table 6-10.4. Surrogate

Analyte	Accuracy Limits (%R)
Toluene-d8	70 - 130

 $[\]mathcal{O}$ %RSD $\leq 30\%$ for all compounds except two compounds not to exceed 40% RSD.

[@] % $R \le 30\%$ for all compounds except two compounds not to exceed 40% R.

^{3 10%} of compounds are allowed to be out but not to exceed $\pm 50\%$.

Table 6-10.5. Summary of Calibration and QC Procedures GC/MS Direct Inject TO-14A/TO-15

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW - 846 tune criteria.	Correct problem then repeat tune.
5 Point Calibration	Prior to sample analysis.	$%RSD \le 30\%$ for all compounds with up to two exceptions $\le 40\%$.	Correct problem then repeat Initial Calibration.
Laboratory Control Standard (LCS)	With each Initial Calibration Curve, prior to sample analysis for each clock.	Recoveries must be within ± 30%D for 90% of the analytes. Outliers must be within 50 – 150%R.	Investigate the problem and if warranted, analyze a new analytical curve.
Continuing Calibration Verification (CCV)	At the start of each analytical batch.	$%D \le 30\%$ with two exceptions up to 40%.	Perform maintenance and repeat tests, or re-calibrate instrument.
Laboratory Blank	After the standards and before samples.	Results less than the laboratory reporting limit.	Inspect the system and reanalyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for the blanks and samples must be within \pm 0.33 minutes of the RT in the daily Method Spike. The IS area must be within \pm 40% of the CCV's IS area, for the blanks and samples.	Blanks: Inspect the system and re-analyze the blank. Samples: Re-analyze the sample. If the IS is within limits in the re-analysis, report the second analysis. If IS is out of limits a second time, dilute the sample until IS is within limits and narrate.
Surrogate	As each standard, blank, and sample is being loaded.	70 - 130%R.	For blanks: Inspect the system and re-analyze the blank. For samples: Re-analyze sample unless obvious matrix interference is documented. If the %R is within limits in the reanalysis, report the second analysis. If %R is out-of-limits a second time, report data from first analysis and narrate.
Laboratory Duplicates	10% of the samples.	RPD \leq 25% for compounds detected at $>$ 5X reporting limit.	Re-analyze the sample for a third time. If it fails again, investigate the cause and correct problem. Otherwise, narrate the non - conforming event.

6.11 ASTM D-1945 - FIXED GASES

This method involves GC analysis of landfill gas, ambient air, or stack gas collected in SummaTM canisters, Tedlar bags, or any vessel that has been demonstrated to be clean and leak free. Samples are analyzed for Methane and fixed gases and can be used to speciate individual light hydrocarbons up to C6. This method is also used to determine caloric content of the gas. Because the sample is withdrawn from the vessel by

positive pressure, rigid containers are first filled to positive pressure using UHP Helium or Nitrogen. Samples are then analyzed using a GC equipped with a FID and a Thermal Conductivity Detector (TCD).

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-11.1. Summary of Method Modifications

Requirement	ASTM D-1945	Air Toxics Ltd. Modifications
Sample Injection Volume	0.50 mL to achieve Methane	1.0 mL.
	linearity.	
Reference Standard	Concentration should not be <	A minimum 3-point linear
	half of nor differ by more than	calibration. The acceptance
	2X the concentration of the	criterion is RSD ≤15%. All target
	sample. Run 2 consecutive	analytes must be within the linear
	checks; must agree within 1%.	range of calibration (with the
		exception of O ₂ , N ₂ , and C6+
		Hydrocarbons).
Sample Analysis	Equilibrate samples to 20-50° F.	Heating of samples
	above source temperature at	is not performed.
	field sampling.	
Sample Calculation	Response factor is calculated	Peak areas are used for all
	using peak height for C5	target analytes to quantitate
	and lighter compounds.	concentrations.
Normalization	Sum of original values	Sum of original values may range
#	Should not differ from	between 85-115%; normalization
	100.0% by more than 1.0%.	of data not performed.

Table 6-11.2. ASTM Modified Method D-1945 Standard Analyte List

	200.00	Acceptance Criteria			
Analyte	RL (%)	Initial Calibration (%RSD)	CCV/LCS (%R)	Precision (%RPD)	
Carbon Dioxide	0.01	≤ 15%	85 – 115	≤ 25%	
Carbon Monoxide	0.01	≤ 15%	85 – 115	≤ 25%	
Ethene	0.001	≤ 15%	85 – 115	≤ 25%	
Ethane	0.001	≤ 15%	85 – 115	≤ 25%	
Acetylene	0.001	≤ 15%	85 – 115	≤ 25%	
Isobutane	0.001	≤ 15%	85 – 115	≤ 25%	
Methane	0.0001	≤ 15%	85 – 115	≤ 25%	
n-Butane	0.001	≤ 15%	85 – 115	≤ 25%	
Neopentane	0.001	≤ 15%	85 – 115	≤ 25%	
Isopentane	0.001	≤ 15%	85 – 115	≤ 25%	
n-Pentane	0.001	≤ 15%	85 – 115	≤ 25%	
Nitrogen*	0.10	≤ 15%	85 – 115	≤ 25%	
NMOC (C6+)	0.01	≤ 15%	85 – 115	≤ 25%	
Oxygen	0.10	≤ 15%	85 – 115	≤ 25%	
Propane	0.001	≤ 15%	85 – 115	≤ 25%	
Hydrogen	0.01***	≤ 15%	85 – 115	≤ 25%	
Helium	0.01**	≤ 15%	85 - 115	≤ 25%	

^{*} For samples that have been pressurized with N_2 , the amount of N_2 in the sample is determined by subtraction.

Table 6-11.3. Summary of Calibration and OC Procedures ASTM Modified Method D-1945

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial	Prior to Sample	ICAL criteria in Table	Correct problem, then repeat
Calibration	Analysis.	6-11.2.	Initial Calibration.
Curve (ICAL)			
Independent	Once per analytical	LCS criteria in Table 6-	Check the system and re-
Source Check	batch.	11.2.	analyze the standard. Re-
Verification			calibrate the instrument if the
(LCS)	·		criteria cannot be met.

^{**} Included by special request only.

^{***} RL is 1.0% when sample is pressurized with Helium.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing	Daily prior to sample	CCV criteria in Table	Check the system and re-
Calibration	analysis and after every	6-11.2.	analyze the standard. Re-
Verification	20 samples or at the		calibrate the instrument if the
(CCV)	end of the analytical		criteria cannot be met. If the
	batch.	'	closing CCV fails, the system
			is checked and the standard is
			re-analyzed. If the second
			analysis fails, identify and
			correct the problem, then re-
			analyze all samples since the
	·		last acceptable CCV.
Laboratory	Daily.	Results less than the	Inspect the system and
Blank		laboratory RL.	troubleshoot until the system is
			free of contamination.
Sample	10%.	RPD ≤25% for	Re-analyze the sample a third
Duplicates		detections >5 times the	time. If the limit is exceeded
		RL.	again, investigate the cause and
			bring the system back to
			working order. If no problem
		G.	is found with the system,
			narrate the data.

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6.12 ASTM D-1946 - ATMOSPHERIC GASES

This method involves GC analysis of landfill gas, ambient air, or stack gas collected in SummaTM canisters, Tedlar bags, or any vessel that has been demonstrated to be clean and leak free. Samples are analyzed for Methane, fixed gases, and Non-Methane Organic Carbon (NMOC) using ASTM D-1946 protocols. Because the sample is

withdrawn from the vessel by positive pressure, rigid containers are first filled to positive pressure using UHP Helium or Nitrogen. Samples are then analyzed using a GC equipped with a FID and a TCD.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-12.1. Summary of Method Modifications

Requirement	ASTM D-1946	Air Toxics Ltd. Modifications
Calibration	A single point calibration is performed using a reference standard closely matching the composition of the unknown.	A minimum 3-point calibration curve is performed. Quantitation is based on a daily calibration standard, which may or may not resemble the composition of the associated samples.
Reference Standard	The composition of any reference standard must be known to within 0.01 mol % for any component.	The standards used by Air Toxics Ltd. are blended to a ≥ 95% accuracy.
Sample Injection Volume	Components whose concentrations are in excess of 5 % should not be analyzed by using sample volumes greater than 0.5 mL.	The sample container is connected directly to a fixed volume sample loop of 1.0 mL. Linear range is defined by the calibration curve. Bags may be loaded by vacuum or by positive pressure.
Normalization	Normalize the mole percent values by multiplying each value by 100 and dividing by the sum of the original values. The sum of the original values should not differ from 100% by more than 1.0%.	Results are not normalized. The sum of the reported values can differ from 100% by as much as 15%, either due to analytical variability or an unusual sample matrix.
Precision	Precision requirements established at each concentration level.	Duplicates should agree within 25 % RPD for detections >5 X's the RL.

Table 6-12.2. ASTM Modified Method D-1946 Standard Analyte List

Compound	RL (%)	ICAL Criteria (%RSD)	LCS Criteria (%R)	CCV Criteria (%D)	Precision Limits (RPD)
Carbon Dioxide***	0.010	≤ 15%	85 – 115	±15	≤ 25%
Carbon Monoxide***	0.010	≤ 15%	85 – 115	±15	≤ 25%
Methane	0.00010	≤ 15%	85 – 115	±15	≤ 25%
Ethene*	0.0010	≤ 15%	85 – 115	±15	≤ 25%
Ethane*	0.0010	≤ 15%	85 – 115	±15	≤ 25%
Nitrogen	0.10	≤ 15%	85 – 115	±15	≤ 25%
NMOC (C2+)	0.010	≤ 15%	85 – 115	±15	≤ 25%
Oxygen	0.10	≤ 15%	85 – 115	±15	≤ 25%
Hydrogen*	0.010**	≤ 15%	85 – 115	±15	≤ 25%

^{*} Ethene, Ethane and Hydrogen are included by special request only.

Table 6-12.3. Summary of Calibration and OC Procedures ASTM Modified Method D-1946

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Curve (ICAL)	Prior to Sample analysis.	RSD ≤ 15%.	Correct problem then repeat Initial Calibration.
Second Source Verification (LCS)	All analytes - once per Initial Calibration, and with each analytical batch.	%R 85 – 115	Check the system and re- analyze the standard. Re- calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis and after every 20 samples.	%R 85 – 115 %.	Check the system and re- analyze the standard. Re- calibrate the instrument if the criteria cannot be met.
Laboratory Blank (He) (N ₂ for He and H ₂ analysis)	Immediately after each daily check standard and prior to sample analysis, or when contamination is present.	Results < RL.	Inspect the system and reanalyze the Blank.
End Check	At the end of analytical sequence. It can be primary (CCV) or second source (LCS).	%R 85 – 115 %.	Check system and reanalyze the standard. If the 2 nd analysis fails, correct the problem. Reanalyze all samples since the last acceptable CCV.
Sample Duplicates	10% of the samples.	RPD \leq 25 % for detections $>$ 5 X's the RL.	Re-analyze the sample a third time. Correct the problem. If no problem is found, narrate.

^{**} RL is 1.0 % when sample is pressurized with He.

^{***} RL can be lowered to 0.001% using a Nickel catalyst and reporting from the FID by special request.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Chromatographic Resolution of CH ₄ from CO (FID)	As needed.	< 50 % valley.	Re-condition the molecular sieve column at similar levels.
Response of CO And CO ₂ (FID)	As needed.	< 30 %.	Re-pack the tube with fresh catalyst and allow to stabilize.

6.13 ASTM D-5504 - SULFUR COMPOUNDS

This method involves GC analysis of whole air samples collected in Tedlar bags. Samples are analyzed for reduced sulfur compounds using ASTM D-5504 protocols using a Sulfur Chemiluminescence Detector (SCD).

ASTM D-5504 is not a prescriptive method therefore modification documentation is not necessary.

Table 6-13.1. ASTM Modified Method D-5504 (Sulfur Compounds) Standard Analyte List

	RL	Acceptance Criteria		
Analyte	(ppbv)	ICAL Ø (% RSD)	LCS/ CCV @ (% R)	Precision (% RPD)
2,5-Dimethylthiophene	4.0	≤ 30	70 -130	≤ 25
2-Ethylthiophene**	4.0	≤ 30	70 -130	≤ 25
3-Methylthiophene	4.0	≤ 30	70 -130	≤ 25
Carbon Disulfide	4.0	≤ 30	70 -130	⁻ ≤ 25
Carbonyl Sulfide	4.0	≤ 30	70 -130	≤ 25
Diethyl Disulfide	4.0	≤ 30	70 -130	≤ 25
Diethyl Sulfide**	4.0	≤ 30	70 -130	≤ 25
Dimethyl Disulfide	4.0	≤ 30	70 -130	≤ 25
Dimethyl Sulfide	4.0	≤30	70 -130	≤ 25
Ethyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
Ethyl Methyl Sulfide	4.0	≤30	70 -130	≤ 25
Hydrogen Sulfide	4.0	≤ 30	70 -130	≤ 25
Isobutyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
Isopropyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
Methyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
n-Butyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
n-Propyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
tert-Butyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
Tetrahydrothiophene	4.0	≤ 30	70 -130	≤ 25
Thiophene	4.0	≤ 30	70 -130	≤ 25

 $[\]mathcal{O}$ Average %RSD \leq 30%, not to exceed 40% for any individual compounds. H_2S %RSD must be \leq 30%.

② Up to 10% allowed to exceed %R criterion (not to exceed $\pm 50\%$); end check may have 20% exceed criterion. All compounds must be within %R limit for short list (five compounds or less)

^{**} Compounds co-elute

6-13.2. Summary of Calibration and QC Procedures for Modified ASTM Method D 5504

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Min of 3 or more	Prior to sample	$RSD \le 30\%$ (average).	
points Calibration		H_2S must be $\leq 30\%$.	repeat canoration.
(ICAL)	difaly 515.	All others must be $\leq 30\%$.	
(IC/IL)		All others must be \leq 40%.	
C 1.0	TT7'/1 /1 T '/' 1	<u> </u>	Cl. 1 d
Second Source	With each Initial	70 - 130 % of the	Check the system, re-prepare
Verification	Calibration; with	expected values for at	and/or re-analyze standard. Re-
(LCS)	each analytical batch.	least 18 of the 20	calibrate instrument if criteria
, , , , , , , , , , , , , , , , , , ,		target compounds.	cannot be met.
		H ₂ S must be within	
		±30%. Recovery	
		< 50% or > 150% will	
		require corrective	
		action. If less than five	
	w.	compounds, all	
		compounds must meet	
		criteria.	
Continuing	Daily prior to sample	Full list: % R for at	Check the system and re-
Calibration	analysis.	least 18 out of the 20	analyze the standard. If the 2 nd
Verification		compounds within 70	analysis fails, identify and
(CCV)		-130 %. H ₂ S must	correct the problem. Corrective
		meet limits.	action may include re-analysis
		Short list: 5	of affected samples out of Hold
		compounds or less, %	Time per client request.
		R for all compounds	
		within $70 - 130 \%$.	
		5-19 compounds,	
		%R for 1 compound	
		or 10% of the	·
		compounds allowed	
		out. H ₂ S must meet	
		limits.	
Laboratory Blank	In between analysis	Results less than the	Inspect the system and re-
	of standards and	laboratory Limit of	analyze the blank. If the third
	project samples.	Quantitation.	blank still has contamination,
,			consult a Scientist or Team
			Leader.
End Check	At the end of the	Recoveries within	Check system and re-analyze
	analytical sequence.	70 - 130% with 20%	the standard. If the 2 nd analysis
		(4 target analytes)	fails, identify and correct the
		allowed out.	problem. Corrective action
•			may include re-analysis of
			affected samples out of Hold
			Time per client request.
Laboratory	10% of the samples.	$RPD \le 25 \%$ for	Re-analyze the sample a third
•		1	time. If the limit is exceeded
Laboratory Duplicates	10 % of the samples.	$RPD \le 25 \%$ for detections $> 5X$ LOQ.	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
			again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate
			the data.

6.14 25C/3C - NON-METHANE ORGANIC CARBON (NMOC) AND FIXED GASES

This method involves GC analysis of landfill gas samples collected in SummaTM canisters. Tedlar bags are not permitted by the method. If clients use Tedlar bags, this modification will be included in the narrative. Samples are analyzed for NMOC as Carbon by

Method 25C using a FID, after an oxidation/reduction process.

For Carbon Dioxide, Methane, Nitrogen, and Oxygen samples are analyzed by Method 3C using a TCD. Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-14.1. Summary of Method Modifications

Requirements	EPA Method 25C	Air Toxics Ltd. Modifications
Calibration Standard Concentrations	Specific levels listed in the method.	Concentrations variable, see SOP.
Column	Carbosieve & Unibeads Separation Columns.	AT1, AT2, & AT3 columns.
System Performance Check Standards	Toluene, Hexane, & Methanol.	Toluene, Hexane, & Acetone.
Oxidation Catalyst	OD Inconel tubing packed with 19% chromia on alumina pellets.	Supplied by Lotus Consulting.

Table 6-14.2. Method 25C Standard Analyte List

Analyte		Acceptance Criteria			
	RL (ppmvC)	ICAL (% D for each level from overall mean)	LCS (% R)	CCV (% R)	Triplicate Injection (% RSD)*
NMOC	50	± 2.5	75 - 125	90 - 110**	≤ 5*

^{*} Triplicate injection RSD criterion is $\leq 2\%$ for the Method 25C initial calibration standards that are run in triplicate.

Table 6-14.3. Method 3C Standard Analyte List

	RL	Low Point		eria		
Analyte	(%)	of Curve (%)	ICAL LCS CCV (% RSD) (% R) (% R)			
Carbon Dioxide	0.10	0.10	≤ 25	75 - 125	85 - 115	
Methane	0.10	0.10	· ≤ 25	75 - 125	85 - 115	
Nitrogen	0.10	0.10	≤ 25	75 - 125	85 - 115	
Oxygen	0.10	0.10	≤ 25	75 - 125	85 - 115	

Table 6-14.4. Summary of Calibration and QC Procedures for Method 25C/3C

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Performance Test	Prior to placing system in operation, after any shut down of six months or more, and after any major modifications.	Oxidation Catalyst Efficiency: Average FID recovery of 3 Methane injections < 1% of standard injected. Reduction Catalyst Efficiency: %D ≤5% for average FID Methane recovery of 3 injections. Column Efficiency Performance: NMOC within ±5% of expected value for Toluene, Hexane, and Acetone standards.	Perform system maintenance then repeat test.
Initial Calibration (minimum of 3 points)	Prior to sample analysis.	3C: $\%$ RSD ≤ 25 25C (NMOC): $\%$ D $\le 2.5\%$ of average RF for each level of Propane standard from overall mean RF, and $\%$ RSD ≤ 2.0 for each triplicate injection.	Correct problem then repeat Initial Calibration.

^{**} For Method 25C (modified) single injection run, CCV's %D ±15%.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS)	With each initial calibration, and with each analytical batch.	±25% of the expected value.	Check system and re-analyze the standard. If the 2 nd analysis fails, identify and correct the problem prior to analysis of samples. Corrective action performed may include re-making the standard and re-calibration of the instrument.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, and every 20 samples.	3C: %D ≤ 15%. 25C: Average %D ≤ 10%, triplicate RSD ≤ 5%. Mod. 25C: %D ≤15% single injection.	Check the system and re- analyze the standard. Perform maintenance and re-calibrate if criteria cannot be met. If closing CCV fails, re-analyze all samples back to the last acceptable CCV.
Laboratory Blank	Following analysis of standards.	Results less than the laboratory RL.	Inspect the system and reanalyze the laboratory blank.
Laboratory Replicates	Triplicate analysis for all NMOC (25C) injections.	RSD ≤ 5%.	Re-analyze the sample a 4 th time. If the limit is exceeded again, narrate the data.
Laboratory Duplicates	10% of 3C samples.	RPD ≤25% for detections >5 times the RL.	Re-analyze the sample a 3 rd time. If the criterion still is not met, the cause is investigated and the system brought back to working order. If no problem is found on the system, the data is narrated.
Laboratory Replicates	Duplicate analysis for 3C samples.	5% Precision.	If the 5% duplicate precision requirement is not met, analyze samples for the 3 rd time. Narrate the discrepancy if the precision is not met after the 3 rd analysis.

6.15 RSK-175 - GC HEADSPACE EQUILIBRATION TECHNIQUE

This method involves GC analysis of the dissolved gases in water samples collected in 40 mL VOA vials. In the laboratory, an aliquot of the sample is injected into a Nitrogen-purged vial and placed into a headspace autosampler where each sample is shaken and heated prior to injection. The autosampler then injects an aliquot of headspace onto a gas chromatographic

column where the gaseous components are separated and detected by a FID or TCD. Analysis is conducted for analytes listed in Table 6-15.2.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-15.1. Summary of Method Modifications

Variance	EPA SOP RSK-175	Air Toxics Ltd. SOP
Sample Collection	Collect sample in 60 mL crimp-top vial.	Collect sample in 40 mL VOA vial. Or pressurized sample cylinder.
Headspace Generation	Headspace is generated in 60 mL sample vial by displacing volume of liquid with Helium. The amount of liquid should be 10% of sample volume in bottle, up to 10 mL.	5.0 mL of sample is displaced with 5.0 mL Nitrogen and transferred to a Nitrogen purged and capped autosampler vial. Headspace is then generated in the auto-sampler vial.
Headspace Injection	Syringe injection of 300 μL headspace into GC.	Autosampler pressurizes sample to fill 1.0 mL loop with headspace sample.
Calibration and Quantitation	Direct injections of gas phase standards are used to obtain a Calibration Curve. Henry's Law is used to calculate mg of gas per Liter of water. Calculation requires recording total volume of serum bottle and headspace, and sample temperature.	Calibration is obtained by spiking headspace samples with gas phase analyte and analyzing using the same procedure as the samples. Quantitation of samples is directly obtained using the Calibration Curve that relates µg analyte/mL water sample to peak area.
Initial Calibration Curve (ICAL)	Linear regression.	Average Response Factor.
Lab Blanks	Blank subtraction is performed.	No blank subtraction; Lab Blank must be less than the Reporting Limit.

Table 6-15.2. RSK-175 Headspace by GC Standard Analyte List

	RL	Acceptance Criteria		
Analyte		ICAL (% RSD)	LCS/ CCV (% R)	Precision (% RPD)
Methane	0.005	≤ 30	70 - 130	≤ 25
Ethane	0.01	≤ 30	70 - 130	≤ 25
Ethene	0.01	≤ 30	70 - 130	≤ 25
Propane	0.001	≤ 30	70 - 130	≤ 25
Propene	0.001	≤ 30	70 - 130	≤ 25
Carbon Dioxide	1.0	≤ 30	70 - 130	≤ 25

Table 6-15.3. Summary of Calibration and QC Procedures for RSK-175 Headspace by GC

QC Check	Minimum Frequency	Criteria	Corrective Action
Five Point Calibration (ICAL)	analysis.	$\%$ RSD \leq 30.	Repeat the calibration.
Second Source Verification (LCS)	With each Initial Calibration; and analytical batch.	70 – 130 % of the expected value.	Check the system and re- analyze the standard. Re- calibrate if necessary.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, and after every 20 samples.	70 – 130 % of the expected value.	Check the system and reanalyze the standard. Recalibrate if necessary.
Laboratory Blank	In between analysis of standards and project samples.	Results less than the laboratory Limit of Quantitation.	Inspect the system and reanalyze the Laboratory Blank.
Matrix Spike (MS)	By client request.	– 150 %.	If unopened sample vial remains, re-prepare MS. If none available, flag result and narrate.
Matrix Spike Duplicate (MSD)	By client request.	RPD ≤ 25%.	If unopened sample vial remains, re-prepare MSD. If none available, narrate.
End Check	At the end of analytical sequence.		Check system and re-analyze the standard. If the 2 nd analysis fails, identify and correct the problem. All suspect samples are reprepared from the available hold vials and analyzed.
Laboratory Duplicates	10% of the samples.	RPD $\leq 25\%$ for Detections $> 5X$ LOQ.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system,

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	
			narrate the data.	
Preparation LCS	With each batch up to 20 samples.	70 – 130% of the expected value.	Re-prepare and analyze the LCS along with associated samples if hold vials are available. If not, flag and narrate the data.	

6.16 TO-17 VOLATILE ORGANIC COMPOUNDS

This method is an alternative to the canister based sampling and analysis methods that are presented in EPA Compendium Methods TO-14 and TO-15. Samples are collected by drawing a volume of air through a sorbent packed tube. The sample cartridges are thermally desorbed by heating and purging with organic-free Helium. The resulting gaseous effluent is then bubbled through 5 ml of organic free reagent grade water and trapped on the sorbent trap of the purge and trap system. The trap is then thermally desorbed for GC/MS analysis.

The procedures in this method outline the use of EPA Method TO-17 protocols to determine the concentrations of volatile organic compounds in air samples collected on sorbent tubes.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6-16.1. Summary of Method Modifications

Requirements	EPA Method TO-17	Air Toxics Ltd. Modifications
ICAL RRF %RSD Acceptance Criteria	≤ 30 % RSD, with two compounds allowed out to 40%RSD.	30 % RSD for 90% of the analyte list. No outliers to exceed 40%.
IS Recoveries	Within 40 % of mean over ICAL (blanks), and w/in 40 % of daily CCV (samples).	Within 40 % of CCV recoveries for blank and samples.
Daily CCV	70 – 130 %.	30 % D for 90% of the analyte list. No outliers to exceed 40%.
Method Blank	Cartridges from the same media batches as the samples. Do not dry purge Lab Blanks.	Cartridges used for daily method blank may or may not be from the same batch or sampling media. Lab Blanks are dry purged to eliminate the possibility of sample anomaly attributed to Dry purge process.
Sorbent Tube Storage	After conditioning of sorbent tubes, wrap the sealed tubes in uncoated aluminum foil and place the tubes in clean opaque container.	After conditioning of sorbent tubes, the sealed sorbent tubes are placed in clean airtight glass culture tubes containing activated charcoal. Sorbent batch certification results confirm the sorbent integrity and storage process.
Sample desorption	Method involves primary and secondary desorption.	After primary desorption, the stream of effluent gas is passed through 5ml of clean purged D.I. water before the secondary desorption. D.I. water acts as a filter for excessive acidic moisture in the samples.

Table 6-16.2. TO-17 Standard Analyte List

Table 6-10.2. 10-1/ Standard A	RL	Acceptance Criteria		
Analytes	(ng)	ICAL (%RSD)	LCS (% R)	(%D)
1,1,1-Trichloroethane	10	30	70 – 130	30
1,1,1,2-Tetrachloroethane	10	30	70 – 130	30
1,1,2,2-Tetrachloroethane	10	30	70 – 130	30
1,1,2-Trichloroethane	10	30	70 – 130	30
1,1-Dichloroethane	10	30	70 – 130	30
1,1-Dichloroethene	10	30	70 – 130	30
1,1-Dichloropropene	10	30	70 – 130	30
1,2,3-Trichlorobenzene	50	30	70 – 130	30
1,2,3-Trichloropropane	10	30	70 – 130	30
1,2,4-Trichlorobenzene	50	30	70 – 130	30
1,2,4-Trimethylbenzene	10	30	70 – 130	30
1,2-Dibromo-3-chloropropane	50	30	70 – 130	30
1,2-Dichlorobenzene	10	30	70 – 130	30
1,2-Dichloroethane	10	30	70 – 130	30
1,2-Dichloropropane	10	30	70 – 130	30
1,3,5-Trimethylbenzene	10	30	70 - 130	30
1,3-Butadiene	50	30	50 – 150	30
1,3-Dichlorobenzene	10	30	70 – 130	30
1,3-Dichloropropane	10	30	70 - 130	30
1,4-Dichlorobenzene	10	30	70 – 130	30
2,2-Dichloropropane	10	30	70 – 130	30
2-Chloropropane	10	30	70 – 130	30
2-Chlorotoluene	10	30	70 – 130	30
Allyl chloride	10	30	70 – 130	30
4-Chlorotoluene	10	30	70 – 130	30
Acrylonitrile	10	30	70 – 130	30
Benzene	10	30	70 – 130	30
Bromobenzene	10	30	70 – 130	30
Bromochloromethane	10	30	70 – 130	30
Bromodichloromethane	10	30	70 – 130	30
Bromoform	10	30	70 – 130	30
Bromomethane	10	30	50 – 150	30
Butylbenzene	10	30	70 – 130	30
Carbon Disulfide	10	30	70 – 130	30
Carbon Tetrachloride	10	30	70 – 130	30
Chlorobenzene	10	30	70 – 130	30
Chloroethane	10	30	50 – 150	30
Chloroform	10	30	70 – 130	30
Chloromethane	10	30	50 – 150	30
cis-1,2-Dichloroethene	10	30	70 - 130	30
cis-1,3-Dichloropropene	10	30	70 – 130	30
cis-1,4-Dichloro-2-butene	50	30	70 – 130	30

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Analytes	RL (ng)	ICAL	LCS		
	(ng)	(%RSD)	(% R)	(%D)	
Cumene	10	30	70 - 130	30	
Dibromochloromethane	10	30	70 - 130	30	
Dibromomethane	10	30	70 – 130	30	
Dichlorodifluoromethane	10	30	50 – 150	30	
Ethylbenzene	10	30	70 – 130	30	
Ethylene Dibromide	10	30	70 – 130	30	
Freon 11	10	30	70 – 130	30	
Freon 113	10	30	70 – 130	30	
Hexachlorobutadiene	50	30	70 – 130	30	
Hexane	10	30	70 – 130	30	
Iodomethane	10	30	70 – 130	30	
Methylene Chloride	10	30	70 – 130	30	
Methyl t-butyl ether (MTBE)	10	30	70 – 130	30	
Naphthalene	50	30	70 – 130	30	
m,p-Xylene	10	30	70 – 130	30	
o-Xylene	10	30	70 – 130	30	
p-Cymene	10	30	70 – 130	30	
Propylbenzene	10	30	70 – 130	30	
sec-Butylbenzene	10	30	70 - 130	30	
Styrene	10	30	70 – 130	30	
tert-Butylbenzene	10	30	70 – 130	30	
Tetrachloroethene	10	30	70 – 130	30	
Toluene	10	30	70 – 130	30	
trans-1,2-Dichloroethene	. 10	30	70 – 130	30	
trans-1,3-Dichloropropene	10	30	70 – 130	30	
trans-1,4-Dichloro-2-butene	50	30	70 – 130	30	
Trichloroethene	10	30	70 – 130	30	
Vinyl Bromide *	50	30	50 – 150	30	
Vinyl Chloride	10	30	50 – 150	30	

^{*} Independent Source Verification Check not available for this compounds.

Table 6-16.3. Internal Standard Recovery Limits Limits

Analyte	CCV IS (%R)	Sample IS
1,4-Dichlorobenzene-d ₄	50 - 200	60 – 140
Chlorobenzene-d ₅	50 - 200	60 - 140
Fluorobenzene	50 - 200	60 – 140

Table 6-16.4. Surrogate Recovery

Analyte	Accuracy (%R)
1,2-Dichloroethane-d ₄	70 - 130
4-Bromofluorobenzene	70 – 130
Dibromofluoromethane	70 - 130
Toluene-d ₈	70 – 130

Table 6-16.5 Summary of Calibration and QC Procedures for Method TO-17

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW - 846 tune criteria.	Correct problem then repeat tune.
5-Point Calibration	Prior to sample Analysis.	%RSD \leq 30% for 90% of the analyte list not to exceed 40%.	Correct problem then repeat Initial Calibration Curve.
LCS	After each initial Calibration Curve and daily prior to analysis.	compounds are allowed	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	$\%D \le 30\%$ for 90% of the analyte list not to exceed 40%.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5-point Calibration Curve.
Laboratory Blank	After the CCV.	Results less than the RL.	Inspect the system and re-analyze the Blank.
Internal Standard (IS)	As each standard, Blank, and sample is being loaded.	CCVs: area counts 50% - 200%, RT w/in 30 sec of mid-point in ICAL.	CCV: inspect and correct system prior to sample analysis. Blanks: inspect the system and reanalyze the Blank.
		Blanks and samples: Retention time (RT) must be within ±0.33 minutes of the RT in the CCV. The IS area must be within ±40% of the CCV's IS area for the Blanks and samples.	Samples: samples cannot be reanalyzed due to the nature of the sorbent cartridges. However investigate the problem by reviewing the data. If necessary, run a Lab Blank to check the instrument performance. Report the data and narrate.
Surrogates	As each standard, Blank, and sample is being loaded.	70 – 130%.	For blanks: inspect the system and re-analyze the Blank. For samples: samples cannot be re-analyzed due to the nature of sorbent cartridges. However investigate the problem by
			reviewing the data. If necessary, run a Lab Blank to check the instrument performance. Report the data and narrate the problem.

6.17 ANALYSIS OF C2-C5 HYDROCARBONS BY GC/FID

This section describes the use of modified EPA Method TO-14A to determine the concentration of Highly Reactive Volatile Organic Compounds (HRVOC) in air using an evacuated Silonite or Summa canister, or a Tedlar bag.

Up to 40 mL of sample is withdrawn from a canister or Tedlar bag using a gas-tight syringe, and is injected directly into a sorbent based focusing interface. The sample is then flash-heated through a dehumidification system that removes background moisture from the sample stream.

The sample stream is then split into two directions: (1) Analysis of Volatile Organic Compounds by full scan GC/MS mode, (2) Analysis of C2-C5 hydrocarbons by

GC/FID. In one analytical run, this configuration is capable of analyzing individual volatile organic compounds as well as C2-C5 hydrocarbons. Since each application is independently calibrated, procedures, QC requirements, and method modifications are referenced in Section 6.10 for TO-14A/TO-15 full scan analysis by GC/MS direct inject. The procedures described in this section are specific for the analysis of C2-C5 hydrocarbons by GC/FID.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Table 6.17-1. Summary of Method Modifications

Requirement	TO-14A	Air Toxics Ltd. Modifications
Sample Drying System	Nafion Drier.	Multi-sorbent.
Sample load volume	400 mL.	Up to 40 mL.
Blank acceptance criteria	< 0.2 ppbv.	< RL.
Dilutions for initial calibration	Dynamic dilutions or static using canisters.	Syringe dilutions, bag dilutions.
Sample collection media	Summa canister.	Air Toxics Ltd. recommends use of summa canisters to insure data defensibility, but will report results from Tedlar bags at client request.
RT window study	Mean +/- 3 X STD within 72 hours.	+/- 0.08 mins (Mean+/-3X STD <0.08).

Table 6.17-2. Modified TO-14A HRVOC by GC/FID Direct Injection (Standard List of Analytes)

		QA Acceptance Criteria		
Analyte	RL (ppbv)	Accuracy Limits (%R)	Precision Limits (Max. RPD)	
Ethane	5.0	70 - 130	≤ 25	
Ethene	5.0	70 - 130	≤ 25	
Propane	5.0	70 - 130	≤ 25	
Propene	5.0	70 - 130	≤ 25	
Acetylene	5.0	70 - 130	≤ 25	
Isobutane	2.0	70 - 130	≤ 25	
Butane	2.0	70 - 130	≤ 25	
trans-2-Butene	2.0	70 - 130	≤ 25	
1-Butene	2.0	70 - 130	≤ 25	
Isobutylene	2.0	70 - 130	≤ 25	
cis-2-Butene	2.0	70 - 130	≤ 25	
Isopentane	2.0	70 - 130	≤ 25	
Pentane	2.0	70 - 130	≤ 25	
1,3-Butadiene	2.0	70 - 130	≤ 25	
NMOC	25	70 - 130	≤ 25	

Table 6.17-3. Summary of Calibration and QC procedures

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3 Point (min) Calibration	Prior to sample analysis.	%RSD ≤ 30%	Correct problem then repeat Initial Calibration.
Lab Control Standard (LCS)	With each ICAL and each analytical batch.	± 30%R.	Investigate the problem and, if warranted, analyze a new analytical curve.
Continuing Calibration Verification (CCV)	At the start and end of each analytical batch, or every twenty samples.	$\%D \le 30\%$.	Perform maintenance and repeat tests, or re-calibrate instrument.
Laboratory Blank	After the standards and before samples.	Results less than the laboratory RL.	Inspect the system and reanalyze the blank. Perform maintenance or report with a B flag.
Laboratory Duplicates	10% of the samples.	RPD \leq 25% for compounds detected at >5X RL.	Investigate and correct problem. Otherwise, narrate the non-conforming event.

6.18 SULFURHEXAFLUORIDE (SF6) BY SINGLE OR DUAL COLUMN GC

This method is generally applied to the analysis of landfill gas, soil gas and industrial and other sources and is typically designed for sub ppbv to ppmv level concentrations. The method involves the use of either an Electron Capture Detector (ECD) or Sulfur Chemiluminescence Detector (SCD) depending on the expected

analyte concentration in the matrix.

Analysis using the ECD will provide a Limit of Quantitation of 0.2 ppbv. It involves direct injection of a 2 mL sample onto dual GC columns configured in series. This assures separation of SF6 from associated hydrocarbons or other interfering material that may be present in the matrix. The SCD method has a Limit of Quantitation of 10 ppbv and involves direct injection of a 1 mL sample onto a single GC column.

Table 6.18-1. Sulfurhexafluoride LOQ and QC

	Low		Acceptance Criteria		
Analyte	RL (ppbv)	Point Of the Curve (ppbv)	(% RSD)	LCS/ CCV/End Check (% R)	Precision (% RPD)
Sulfurhexafluoride (ECD)	0.2	0.2	≤ 30	70 - 130	≤ 25
Sulfurhexafluoride (SCD)	10	10	' ≤ 30	70 - 130	≤ 25

Table 6.18-2. Summary of Calibration and QC Procedures for Sulfurhexafluoride by GC

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Min. Five Point Calibration	Prior to sample analysis.	$\%$ RSD \leq 30.	Repeat the calibration.
Second Source Verification (LCS)	With each Initial Calibration; with each analytical batch.	70 – 130 % of the expected value.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification	Daily prior to sample analysis, and after every 20 samples.	70 – 130 % of the expected value.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.
Laboratory Blank	Daily.	Results less than the laboratory RL.	Inspect the system and re-analyze the Laboratory Blank.
End Check	At the end of analytical sequence.	70 - 130 % of the expected value.	Check system and re-analyze the standard. If the 2 nd analysis fails, identify and correct the problem. All suspect samples are re-analyzed.

6.19 ANALYSIS OF C2-C12 HYDROCARBONS USING PAMS METHOD GC/FID/FID

The method is developed and based on EPA Method 600-R-98/161 (Technical Assistance Document for Sampling and Analysis of Ozone Precursors). This method identifies and quantifies volatile organic compounds (VOCs) that play a critical role in the photochemical formation of ozone in the atmosphere. These non-polar VOCs range in volatility from C_2 to C_{12} and are comprised of aromatics, olefins, and paraffins. Up to 50

mL of sample is introduced into a GC system from a SUMMA canister or a Tedlar bag using a Mass Flow Controller and a vacuum system. A digital meter readout provides a visual indication of the sample flow during sampling. The sample is focused onto an Air Toxics Ltd. designed sorbent-based interface. The concentrated sample is then flash heated through a de-humidification system that removes background moisture from the sample stream. The sample stream is then split into two directions and analyzed by a dual-column and dual-FID detector GC system.

Table 6.19-1 Standard List of Controlled Compounds

Analyte	Accuracy Limits (%R)
Ethane	80-120
Propane	80-120
Propene	80-120
Isobutane	80-120
Butane	80-120
Isobutene or 1-butene	80-120
Isopentane	80-120
n-Pentane	80-120
1-Pentene	80-120
n-Hexane	80-120
Benzene	80-120
n-Octane	80-120
Toluene	80-120
o-Xylene	80-120
n-Decane	80-120

Table 6.19-2. Standard List of Analytes (AL-2O3/KCL Column)

		QA Accepta	QA Acceptance Criteria		
Analyte	RL PpbC (LOQ)	Accuracy Limits (%R)	Precision Limits (Max. RPD)		
Ethane *	10	80-120	≤ 25		
Ethene	10	NA	≤ 25		
Propane*	10	80-120	≤ 25		
Propene*	10	80-120	≤ 25		
Isobutane*	10	80-120	≤ 25		
Acetylene	10	NA	≤ 25°		
Butane*	10	80-120	≤ 25		
Trans-2-butene	10	NA	≤ 25		
1-Butene*	10	80-120	≤ 25		
Cis-2-Butene	10	NA	≤ 25		
Cyclopentane	10	NA	≤ 25		
Isopentane*	10	80-120	≤ 25		
Pentane*	10	80-120	≤ 25		
1,3-Butadiene	10	NA	≤ 25		
Trans-2-Pentene	10	NA	≤ 25		
1-Pentene*	10	80-120	≤ 25		
Cis-2-Pentene	10	NA	≤ 25		
2,2-Dimethylbutane	10	NA	≤ 25		
2,3-Dimethylbutane	10	NA	≤ 25		
Isoprene	10	NA	≤ 25		
2-Methylpentane	10	NA	≤ 25		
3-Methylpentane * controlled analytes	10	NA	≤ 25		

Table 6.19-3. Standard List of Analytes (RTX-1 Column)

		QA Accepta	QA Acceptance Criteria		
Analyte	RL PpbC (LOQ)	Accuracy Limits (%R)	Precision Limits (Max. RPD)		
1-Hexene	10	NA	≤ 25		
Hexane*	10	80-120	≤ 25		
Methylcyclopentane/2,4-	10	NA	≤ 25		
Dimethylpentane					
Benzene*	10	80-120	≤ 25		
Cyclohexane	10	NA	≤ 25		
2-Methylhexane/2,3-Dimethylpentane	10	NA	≤ 25		
3-Methylhexane	10	NA	≤ 25		
2,2,4-Trimethylpentane	10	NA	≤ 25		
Heptane	10	NA	≤ 25		
Methylcyclohexane	10	NA	≤ 25		
2,3,4-Trimethylpentane	10	NA	≤ 25		
Toluene*	10	80-120	< 25		
2-Methylheptane	10	NA	< 25		
3-Methylheptane	10	NA	<u>≤ 25</u>		
Octane*	10	80-120	< 25		
Ethylbenzene	10	NA	<u>≤</u> 25		
m,p-Xylene	10	NA	<u>≤</u> 25		
Styrene	10	NA	<u>≤ 25</u>		
o-Xylene*	10	80-120	≤ 25		
Nonane	10	NA	≤ 25		
Cumene	10	NA	≤ 25		
n-Propylbenzene	10	NA	≤ 25		
m,p-Ethyltoluene	10	NA	≤ 25		
1,3,5-Trimethylbenzene	10	NA	≤ 25		
o-Ethyltoluene	10	NA	≤ 25		
1,2,4-Trimethylbenzene	10	NA	<u>≤ 25</u>		
Decane*	10	80-120	<u>≤ 25</u>		
1,2,3-Trimethylbenzene	10	NA	<u>≤</u> 25		
m-Diethylbenzene	10	NA	<u>≤</u> 25		
p-Diethylbenzene	10	NA	<u>≤</u> 25		
Undecane	10	NA	< 25		

^{*} controlled analytes

		QA Acceptance Criteria		
Analyte	RL PpbC (LOQ)	Accuracy Limits (%R)	Precision Limits (Max. RPD)	
NMOC	20	NA	< 25%	

Table 6.19-4. Summary of Calibration and QC Procedures for PAMS Hydrocarbons

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Pre-measurement Chromatographic system Verification	During initial setup, a minimum of 3 Retention Time standards containing all target VOCs, analyzed over a period of days to establish retention time windows for each components.	RT drift must be less than 0.1 minutes.	Correct the problem, recalibrate the analytical system.
Method Detection Limits	Annually, using 40CFR, Part 136, Appendix B.	Must meet the DQO limit.	Re-evaluate and improve the system. Repeat the study.
ICAL	Prior to sample analysis, and when major system maintenance is performed. Use primary calibration standard, containing propane and benzene. Analyze a minimum of three points to bracket the calibration range. Generate per Carbon response factor for each column.	Correlation coefficient 0.995.	Repeat individual standard analysis. Correct system problem if any. Re-prepare standards and repeat Initial Calibration.
LCS	Contain analytes listed as controlled in Tables 6.19-2 and 6.19-3 Analyzed after each calibration curve.	Recoveries must be within ± 20%D.	Repeat sample analysis. Investigate the problem and if warranted, analyze a new analytical curve.
Continuing Calibration Verification (CCV)	Daily mid-point standard before sample analysis.	RF within 10% RPD of calibration curve average RF.	Repeat sample analysis. Perform maintenance and or re-calibrate instrument.
Retention time standard containing all target VOCs	Daily before sample analysis.	Retention time is used for daily RT calibration. Recovery of Table 1. analytes must be 80-120%.	Repeat sample analysis. Perform maintenance and or re-calibrate instrument.
System Background and Carry-over check (Humid zero air)	Daily. After the standards and before samples, and whenever a sample exceeds the established carryover limit.	Less than 20 ppbC total or less than 10 ppbC per column.	Repeat analysis, check system for leaks, clean system with humid air, condition sample trap. Re- analyze system blank.
Canister cleaning certification	All canisters prior to use.	Less than 10ppbC total.	Re-clean canister and re-certify.
Field Duplicate	10% of field sample.	RPD \leq 25% for compounds detected at >5X RL or per DQO.	None in the Lab.
Laboratory Duplicates	10 % of samples.	RPD \leq 25% for compounds detected at >5X RL or per DQO.	Re-analyze the sample for a third time. If it fails again, investigate the cause and correct problem. Otherwise, narrate the non-conforming event.

7.0 DATA COLLECTION, REVIEW, REPORTING, AND RECORDS

7.1 DATA COLLECTION

All analytical results are generated from the instrument software. Data is acquired using a PC/Windows based platform. Data processing occurs on a UNIX based network system. Desktop PCs configured with HP Chemstation software acquire the sample analysis results. Once the acquisition is complete, a post-run macro automatically transfers the raw data files from the hard drive of the acquisition PC to the UNIX server. The UNIX server is a HP D390 server with a full RAID system. This fault tolerant server is configured to manage hot-swappable hard drives and memory cards to avoid any serious downtime. This server is configured with HP-UX 10.2, Thermo Lab Systems Target Software, and Omniback software.

All sample data is stored and processed on the UNIX server. Access to this server is limited based on the privileges associated with the passwords. users' Only Systems the Administrator and the IT Manager maintain full access to the system (which includes exclusive privilege for the adjustment of acquisition station clock times). The system servers are physically located in a secured office, which is locked during off-hours. The data stored on the UNIX server is backed up nightly, weekly, and monthly using a modified grandfather-father-son (GFS) backup rotation. All permanent backup tapes are stored in a secure fireproof safe.

Data reduction of analytical files is accomplished using Thermo Lab Systems' Target software, which allows for complete traceability of the data results. Additionally, multiple permanent records of the data reduction files are maintained through the data back up procedures, minimizing the threat of any lost data trail evidence. Chemists must

login to the data reduction software using a unique password in order to access and work with the sample data files. Once the chemists have successfully logged-in to the working environment, all of their activities are tracked and logged by the Target software's electronic assessment trail. The assessment trail file is a tamper proof record of each event that occurred with the data file. The assessment history for a data file contains:

- Date of Change
- Time of Change
- Name of User who made the Change
- Parameter Changed
- Old Value
- New Value
- Reason for Change (if applicable)

The assessment trail file is completely secure within the Target software and cannot be modified or deleted by any user. A hardcopy of the sample assessment trail can be provided upon request for specialized data validation packages. Whenever an electronic raw data assessment is requested, the assessment trail file is automatically included.

Once data reduction is complete, the Scientist or Analyst transfers a copy of the sample results file along with all associated batch QC results into the laboratory's SQL database from which reports are ultimately generated.

7.2 DATA REVIEW

Following analysis, the bench chemist verifies that the computer generated data reduction is correct using the Data Review Checklist (Exhibit 7.2). There are five categories of data review performed in the laboratory.

These categories include:

- I. Analytical review performed by the bench reporting chemist. This review includes a review of raw data, verification of all method and project specific QC requirements, the addition of data qualifier flags when needed, and documentation of any unusual circumstances.
- II. Technical review performed by team leader or QA-approved peer e.g., analysts who have demonstrated proficiency. This is the same type of review performed in Category I, however, it may be performed either by the same person that performed the analysis or by a second individual if specified by the project profile.
- III. QA review performed by a quality assurance specialist. This review is similar to that performed in Categories I and II, however is done with an emphasis on overall quality of the data and verification that standard quality assurance systems are functioning. Data integrity surveillance checks are performed at this level.
- IV. Management review by a Director, Department Manager, Team Leader or approved peer. This is a review to ensure the accuracy of the final hardcopy or electronic report. Data integrity surveillance checks are performed at this level.
- V. <u>Electronic deliverable review.</u> This review is performed when electronic data deliverables are requested. This review ensures the accuracy of the final electronic report.

Regardless of the TAT, categories I, II, and IV (or I, II, and V if only electronic reporting is requested) are performed on every data package. As noted earlier analysts who have demonstrated proficiency may perform a category II review. A minimum of 10% of all data packages receive category III review.

Some clients request that 100% of their final data packages undergo an additional review. The review in this case is performed by the team leader, QA-approved peer, or QA personnel. Technical peer review (Category II), must be performed by a different individual than the original analyst, even when that person has the classification of scientist or higher. A request for Technical peer review shall be documented in the project profile.

7.3 FINAL REPORT PRODUCTION

7.3.1 Automatic Data Transfer (ADT) System

Most data reports are created using ADT from the analytical instrument to a custom-reporting module. Approved bench analysts on each team review the raw data at the instrument and then transfer a copy of the sample results file electronically through a network server to the main database. Once in the database, the data results are automatically formatted into predesigned method templates using the reporting module. The method templates are designed at sample login and a review copy is faxed for client approval prior to reporting. Reporting analysts on each analytical team batch samples results with OA results and any additional information for any sample duplicates or reanalysis.

7.3.2 Manual Data Entry

Results that cannot be reported using the ADT system are manually entered into a validated, pre-programmed EXCEL spreadsheet. The final report is thoroughly reviewed by an approved team member.

7.3.3 Report Compilation

Data reports are designed to include all necessary information which would be required for traceability including:

- Analytical laboratory name, address, and phone number
- Name and address of the client
- Project name or number (title)
- Total number of pages
- Sample field I.D. number
- Laboratory I.D. number
- Receipt pressure
- Dates of collection and receipt
- Date of extraction (if applicable)
- Date and time of analysis
- Applicable method reference
- Instrument number
- Analytical run file name
- Analyte list
- Dilution factor
- Reporting Limit
- Amount detected in units specified
- Surrogate percent recovery
- Laboratory Director signature
- Chain-of-Custody Record

Each report contains a comprehensive Laboratory Narrative which describes the number of samples received in that batch, any abnormal receipt conditions, any deviations from method specific hold times, the analytical method used, any modifications taken by the lab to the referenced method, and any deviations from standard protocol experienced during sample receiving and analysis. Expected and unexpected deviations that may occur during the analysis of the samples are contained in template format. The Narrative is unambiguous and clearly defines both the nature and substance of the variation.

The QA Manager is responsible for creating, and publishing the templates on a secured and shared network drive. The laboratory staff copies appropriate portions of the template

into the Laboratory Narrative document. This approach standardizes the language used in the narratives. The narrative is reviewed using the check sheet in Exhibit 7.2.

The final report is compiled in such a fashion that each subsection is unambiguous and inseparable from the body of the report. A unique page number appears on every page of the report. The estimated uncertainty of the test results may be included on the report at client request (see Section 8.4).

After all OC results have been reviewed and any deviations from the acceptance criteria are noted in the Laboratory Narrative section of the report the Laboratory Director. Department Managers, Team/Task Leaders or Scientists who are approved by the QA Department for relevant analytical procedures may apply an electronic signature to the final reports. The electronic signature on the report cover page means that the signatory accepts responsibility accuracy for the completeness of the data generated. The approved signatory corresponds to the Chief Executive Officer. The QA manager keeps a log of the approved applicators of electronic signature to final reports, and ensures that each applicator has the necessary education and experience.

Application of the electronic signature will automatically lock the workorder thus preventing changes to the original report. If amendments are required due to omissions, errors or additional requests a workorder reissue is initiated. All reissues receive a unique workorder number to distinguish them from the original issue. Reissued reports require a reason for the reissue and date of the reissue in the Laboratory Narrative. The laboratory maintains all supporting documentation for the revision including corrections, additions, or deletions relative to the original report.

7.4 Electronic Reporting and Review

The ATLAS Electronic Diskette Deliverable (EDD) software allows the user to create EDDs in the ATL standard format and more complex custom/client-specific formats. The ATLAS EDD software uses the data from the SQL database for these deliverables, while allowing the users to add custom fields when necessary. The laboratory can produce ERPIMS, JEMS, IRDMIS, and COELT deliverables. The ATL standard EDD format is delivered in Excel (.xls) format. Other client-specific formats can also be generated.

7.5 eCVP/EDD and Reporting in Adobe Format or Diskette

eCVP refers to the electronic conversion of laboratory data compiled as a Comprehensive Validation Package in Adobe Acrobat Portable Document Format (.pdf) and archival onto CD-ROM. The eCVP simultaneously meets the requirements of a Level III or IV data validation package. Adobe Corporation's .pdf documents are an exact replica of the original document, but are smaller in file size than the original document format, thereby reducing the amount of storage space required. Adobe Acrobat .pdf provides a convenient way to view and print images at high resolution. The .pdf document is then recorded onto read-only compact discs (CD). The digital information on this type of disc is injection-molded into the substrate against an aluminum reflective coating. The CDs are then archived.

In addition, Electronic Data Deliverables (EDD) can be generated on diskette or as a compressed WinZip file according to client specifications. An EDD is a flat file (i.e., a spreadsheet with data in fields and records) that can be imported easily by most database management system (DBMS) software. Air Toxics' standard EDD fields are summarized in Table 1. The EDD is created in Microsoft Excel® and converted to a comma delimited (.csv) format.

Table 1. ATL DISKETTE DELIVERABLE STANDARD FORMAT

FIELD NAMES	FORMAT	WIDTH
LABSAMPLEID	CHAR	15
LABCODE	CHAR	3
MATRIX	CHAR	3
METHOD	CHAR	10
CLIENTSAMPID	CHAR	. 15
SAMPDATE	DATE	8
ANALDATE	DATE	8
ANALTIME	TIME	4
LABCTLID	CHAR	8
DILUTION	NUMBER	5
REPLMT	NUMBER	5
UNITS	CHAR	4
RESULTS	NUMBER	5
DATAFLAGS	CHAR	. 2
COMPOUND NAME	CHAR	40
CAS#	CHAR	12
COMMENTS	CHAR	50

LABSAMPLEID:

Sample identifier assigned by ATL.

LABCODE:

Laboratory identifier (ATL).

MATRIX:

Sample Matrix.

METHOD:

Analytical method of analysis.

CLIENTSAMPID:

Sample identifier from Chain of Custody.

SAMPDATE:

The date the sample was collected.

ANALDATE:

The date the sample was analyzed.

ANALTIME:

The time the sample was analyzed.

LABCTLID:

Laboratory batch number.

DILUTION:

Dilution factor.

REPLMT:

Detection limit for sample.

UNITS:

Reporting units of measure.

RESULTS:

Parameter value or result.

DATAFLAGS:

Data qualifiers.

COMPOUND NAME:

The name of each compound analyzed.

CAS#:

The CAS registry number for each compound.

COMMENTS:

General comments field.

Exhibit 7.1. Example eCVP Cover Page



COMPREHENSIVE VALIDATION PACKAGE

Modified TO-15 INVENTORY SHEET Work Order #: 0605678

	Page	e Nos.
	From	То
Work Order Cover Page & Laboratory Narrative	1	4
a. Lumen Validation Report		·
2. Sample Results and Raw Data (Organized by Sample)	5	81
a. ATL Sample Results Form		
b. Target Compound Raw Data		
-Internal Standard Area and Retention Time Summary		
-Surrogate Recovery Summary (If Applicable)		
-Chromatogram(s) and Ion Profiles (If Applicable)		
* * * * * * * * * * * * * * * * * * *		
. QC Results and Raw Data	00	
a. Method Blank (Results+ Raw Data)	82	89
b. Surrogate Recover Summary Form (If Applicable)	90	90
c. Internal Standard Summary Form (If Applicable)	91	91
d. Duplicate Results Summary Sheet	**	
e. Matrix Spike/Matrix Spike Duplicate (Results + Raw Data		245
f. Initial Calibration Data (Summary Sheet + Raw Data)	92	245
g. MDL Study (If Applicable)		
h. Continuing Calibration Verification Data (Summary Sheet	246	259
i. Second Source LCS(Summary + Raw Data)	260	278
i. Extraction Logs	**	
k. Instrument Run Logs/Software Verification	279	280
l. GC/MS Tunc (Results + Raw Data)	281	300
Shipping/Receiving Documents		
a. Login Receipt Summary Sheet	301	302
b. Chain-of-Custody Records	303	303
c. Sample Log-In Sheet	304	304
d. Misc Shipping/Receiving Records (list of individual records)		
Sample Receipt Discrepancy Report	305	306
Other Records (describe or list)		
a. Manual Spectral Defense	·	
b. Manual Integrations		**
c. Manual Calculations		
d. Canister Dilution Factors	307	309
e. Laboratory Corrective Action Request		
f. CAS Number Reference	310	311
g. Variance Table		
h. Canister Certification		
i. Data Review Check Sheet	312	312
The probability of the Late of the Constitution of the Constitutio		
Comments:		
Completed by:	,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	
Theresa Laflesh / Docur	nent Control	6/15/06
(Signature) (Print Name &	Title)	(Date)
(Signature) (Fillit Name &	11110)	(Date)

7.6 RECORDS OF METHOD CAPABILITY

Prior to sample analysis, the laboratory must demonstrate the ability to meet method accuracy and precision objectives. accomplished through an initial multi-point calibration, analysis of four consecutive second source check standards. completion of a Limit of Detection (MDL) study. The mean recovery for each target analyte must be within current laboratory generated control limits with regard to test and compound. Following this initial set-up, there continuing requirement for demonstration of method capability any time the equipment undergoes significant change, such as different column phase and different concentrator design. Records of these tests are kept for a period of at least 5 years.

There is also a requirement for personnel involved with sample analysis to demonstrate both initial and on-going proficiency in the specific test method. Staff proficiency is accomplished by analyzing any of the following:

- Analysis of four replicate second source check standards either on the same day or on four separate days
- Successful completion of an independent PT sample (on-going proficiency only)

The demonstration of proficiency is considered acceptable if the accuracy and precision objectives of the test method are met. Demonstration of proficiency must occur at least once per year to be considered current. Personnel may not proceed with sample analysis unless the demonstration of proficiency is current. Documentation of method proficiency is kept in each analyst's training record. Documentation must be kept for a period of 5 years. The 'Demonstration of Certification Capability Statement' is completed each time a demonstration of proficiency study is completed and appears in the analyst's training record along with the relevant raw data summary.

Continuing Demonstration of Proficiency is required on a yearly basis. The scientist/analyst is required to perform 4 consecutive LCS analyses, or if this is not possible due to the nature of the work assignment schedule, such as working second shift, a duplicate analysis paired with another analyst's or scientist's results that demonstrate acceptable %RPD will be acceptable.

7.7 RECORD STORAGE

The laboratory has a system for record storage such that historical reconstruction of all activities can be made. Raw data includes:

- Instrument run logs
- Instrument calibrations
- Data acquisition files
- Assessment trails
- Manual and spreadsheet calculations
- Date of analysis
- Instrument used
- Sample chain of custody
- Analyst initials and date
- Data review checklists
- Corrective action reports

The laboratory also maintains files dealing with client correspondence. The Client Contact database stores the date and time of the contact along with a brief summary of the conversation and any decisions made affecting sample status. When a decision is made to proceed with analysis of compromised samples, the contact is logged into the database and a note is made on the Sample Discrepancy Log. Both the electronic and hardcopy files are maintained for a period of

at least 5 years. Additional project management information stored includes:

- The Project Profile
- Client contact database
- Correspondence relating to sample disposition
- Contracts
- SOWs and/or QAPPs

The laboratory maintains electronic and hardcopy reports, as well as supporting information including calibrations, Limit of Detection (MDL) studies, logbooks, and SOPs for a minimum of 5 years. Records stored on electronic media are supported by both hardware and software necessary for retrieval. If the laboratory changes ownership, then responsibility for file storage transfers to the new entity. If the laboratory were to close its doors entirely, then allowance would be made to return files to those clients who contact the laboratory within 30 days of when notice is given. Under either scenario, the transfer of ownership notice would be provided to clients through the NELAP national database and on the ATL web page.

The record keeping system allows for historical reconstruction of all laboratory activities from sample receipt to reporting. The record system includes:

- The identity of personnel involved in sample receiving, preparation, calibration, and analysis.
- A log of names, initials, and signatures for individuals who are responsible for signing or initialing any laboratory record.
- A unique identifier for each piece of equipment used.
- Initials and date for responsible staff at each step in the analytical process.
- Direct, prompt, and legible manual recording in bound logs using permanent black ink.

- Entries in logs that are not obliterated by erasures, over-writing, or markings. All corrections are made by single line strike out of the error. Each strike out is initialed and dated by the person making the correction. Any items, such as computer generated logs or spreadsheets that are pasted into the bound logbook, have initials and date appearing across the item boundary in such a fashion that removal is apparent.
- Records generated by computer have either hardcopy or write protected back-up copy.
- The QA Department creates and tracks all logbooks throughout their lifetime. Each logbook contains a new logbook request form which is filled out by laboratory personnel and submitted to the QA Department. QA personnel create the logbook by first entering the new logbook into the Inventory Database. This database contains information such as the Book #, its Title, the person's initials and date that created the logbook, the start date, the date it was finished being used and the Location of the logbook. Once the necessary information is entered into the database, the logbook is created and given to either the team/task leader or the person that submitted the request. When the logbook is completed or no longer in use, it is submitted to the QA Department. QA personnel update the Inventory Database with the finished date, the location and the logbook is archived.

7.8 CONFIDENTIALITY OF DATA

The data generated by analyzing a sample is considered to be the property of the entity appearing in the "BILL TO:" field of the work order request unless other contractual arrangements have been made. Accordingly,

that data is treated as confidential information and released only to that client, as identified by associated contractual agreements unless written permission is given to proceed otherwise. All data generated under NELAP related fields of testing shall be made available to recognized agents of any laboratory accrediting authority for purposes inspection and verification during an onsite visit. Clients will not be notified when the accrediting authority reviews data during the normal course of the onsite assessment. Clients will be notified any time a request is made by the accrediting authority to remove copies of sample files, either electronic or hardcopy, from the laboratory. Client written approval must be arranged prior to removal of the files from the laboratory unless the request is accompanied by appropriate court order. Both e-mail and facsimile data are treated as confidential by noting on the cover page:

"The information contained in this communication is confidential and intended only for the use of the individual or entity named above. Any other use, dissemination, distribution, or copying of this communication is prohibited. If you have received this communication in error, please notify us by telephone and return the original message to us via US mail."

Client confidentiality is observed in accordance with guidelines described in NELAC Chapter 5.5.10.6 (2003):

"The laboratory shall ensure that, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will follow documented procedures that ensure the requirements of this Standard are met and that confidentiality is preserved."

Exhibit 7.2.

		m		_	DATA REVIEW CHECKLIST Work Order #:
A			M	Q	Analysis/Reporting vs. Project Profile/SOP requirements checked (i.e. 100% Dups, J-Flag to MDL, etc) The final report has the correct reporting list, special units, and header info.
					Lab Narrative is correct (proper method & description/Receiving & Analytical notes correct)
					Corrective Action issued - #
					LUMEN validation report present and initialed
					Lab Blank, CCV, LCS and DUP met QC criteria Hold time is met for all samples Appropriate data qualifier flags are applied Manual integrations for samples and QC are properly documented Samples analyzed within the project or method specific clock
					Retention times have been verified Appropriate ICAL(s) included At least one result per sample is verified against the target quant sheets/raw data
					Dilution factor correctly calculated (sample load volume, syringe and bag dilutions, can pressurization(s)) Correct amount of sample analyzed (i.e. sample not over-diluted) Spectra verified - documentation of spectral defense included (Section 5A of eCVP pkg) TICs resemble reference spectra TICs between duplicate samples are consistent
					Checked samples for trends (i.e. Influent>Effluent, Landfill or Ambient etc) Special units for all samples in the final report are correctly calculated
	. —				Manually entered results checked (i.e. special CCV compounds)
					TPH/NMOC (verify calculations and correct reference compound used) Chain of Custody scanned correctly Verify sample id's vs. chain of custody
					Samples pressurized w/ appropriate gas $(N_2 \text{ or He})$
					Extra printed copies are provided per client profile Final invoice amount correct (adjusted for TAT, Penalties, Re-issue Charges etc.)
					Client LUMEN report reviewed for accuracy and completeness
Not		o inc <u>A/R</u>		: not	ing samples with QA/QC problems, Blanks with positive hits, narratives, etc.)
		M/Ç);		
(Analy	vtica	A 1 Re	viev	ry/Date) R/T M Q (Reporting Review/Date) (Management Review/Date) (QA Review/Date) R:

8.0 ESTABLISHING ACCEPTANCE CRITERIA

8.1 Control Chart Program

Air Toxics Ltd. complies with guidance from 17025:1999(E), Section 5.9, to ISO/IEC determine quality control limits. This regulation suggests that statistical techniques may be used to detect trends, but does not mandate acceptance or rejection of analytical results based on use of historically derived control limits. Additionally, NELAP does not address the issue of control charting. Therefore, in accordance with ISO/IEC 17025:1999(E). Section 5.9, quality controls are in place to monitor validity of tests and calibrations only.

Historically derived control limits are generated by the QA Department upon client request. Control Limits may be updated less often (or not at all) for methods which are performed so infrequently that it is difficult or impossible to gather at least 20 data points. These limits, however, are not used to validate data unless required by specific client request.

8.2 Establishing Control Limits

Control limits are generated from a minimum of 20 randomly chosen data points. The calculations used to establish and update these investigative limits include:

Upper Control Limit = M +3SLower Control Limit = M -3SUpper Warning Limit = M +2SLower Warning Limit = M -2S

Where:

M: The population mean recovery of at least 20 points, and

S: The standard deviation of the population.

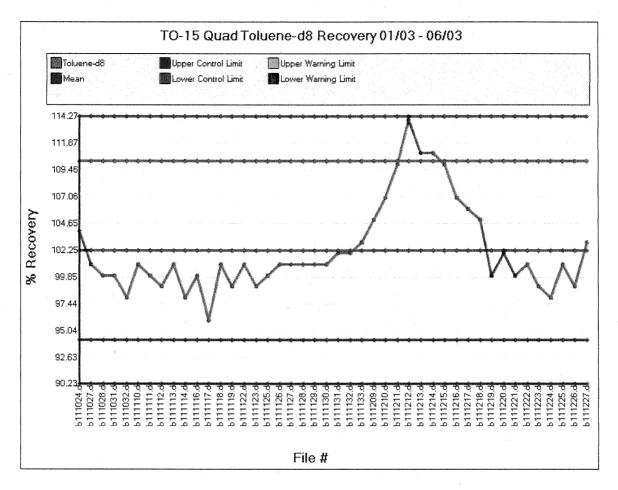
8.3 Interpreting Control Limits

Calculated control limits based on historical data for surrogate and LCS recoveries are used to demonstrate statistical control and display method variability, and are not used to qualify actual sample recoveries. Additionally, control limits may not be representative of the analytical process if less than 20 points are generated for a given method. As a result, Air Toxics Ltd. uses the default limits prescribed for each method in the corresponding SOP and Section 6.0 of this document. Historically derived control limits are used to evaluate LCS or surrogate results only when requested by clients or certifying agencies.

8.4 Measurement Uncertainty

Upon request from a client, Air Toxics Limited will report measurement uncertainty for a given analyte reported by a specific method. Measurement uncertainty is calculated as a function of historical LCS Control Limits (suggested by the American Association For Laboratory Accreditation Interim Policy on Testing Measurement Uncertainty for Laboratories, August 2000). This policy is valid for environmental test methods in which measurement uncertainty is not defined. estimated using standard Uncertainty is deviation of laboratory control samples of more than 50 points. Measurement uncertainty is estimated to the 95% confidence interval and expressed as $\pm 2X$ the standard deviation of the mean percent recovery of each given analyte. If 50 points do not exist from the current control limit calculation, the collection period may be expanded.

Exhibit 8.1. Control Chart



9.0 PREVENTIVE MAINTENANCE

The best form of preventive maintenance is to have good, new stuff and a lot of it. If the customer expectations for quality, turn around time, and price are to be met, then the instrumentation must be maintained in a fashion that supports the quality objective. The program is designed to adequately protect the laboratory from unexpected instrument failure and minimize scheduled instrument down time. Preventive maintenance consists of an on-going program of routine maintenance, service contracts, and a comprehensive inventory of spare parts.

The likelihood of unscheduled down time tends to increase as instrumentation ages. A three year lease term provides nearly optimum instrument life cycles. At the end of the lease term, the equipment will be exchanged for new models.

9.1 ROUTINE MAINTENANCE

The bench analyst monitors instruments for potential failure on a daily basis. The analysis of blanks and control standards at the start of the day and as analysis continues helps to provide real time feedback to the analyst on the condition of the instruments. Routine maintenance, specific to the various types of instruments, is covered in the method SOPs.

Any routine or major maintenance is documented in the bound maintenance logbook assigned to each instrument. The date of the maintenance, work performed, and analyst's initials are included.

If a malfunction occurs and control of the analytical system cannot be demonstrated using the QC parameters, discussed in section 4.3, the instrument is removed from production until analytical control can again be demonstrated.

9.2 SERVICE CONTRACTS

Some analytical systems are covered under manufacturer service agreements. These agreements cover all forms of hardware failure and include regular hardware upgrades as needed. The response time is guaranteed to be within 48 hours under the agreement and includes parts and labor.

Some contracts cover regularly scheduled routine maintenance. Leased instrumentation is similarly covered by service agreements either through the leasing agency or directly with the manufacturer.

In addition, the Technical Services group performs biannual (every six months) preventative maintenance on the mass spectrometers. These records are kept in the individual instrument's maintenance logbooks.

9.3 SPARE PARTS INVENTORY

A normal inventory of analytical consumable parts most frequently required is maintained in the laboratory. These parts are typically not covered by the service agreements and may take several weeks to acquire on an as needed basis. An inventory is required to minimize instrument down time and facilitate routine maintenance. An inventory of design parts is also maintained including:

- Stainless steel valves
- Tubing
- Various connecting nuts and ferrules
- Tools
- Flow controllers
- Flow sensors
- Electrical connectors
- Sheet metal
- Abundance of miscellaneous items
- Multipliers and other MS source parts

The laboratory invests a significant amount of money every year in lab/computer and research supplies.

9.4 Control Of Miscellaneous Monitoring, Measuring, Testing, And Data Collection Equipment

In addition to the equipment used directly in the analysis of samples, ATL uses various other monitoring, measuring, testing, and data collection equipment. This equipment includes: analytical balances and weight sets, pressure gauges, flow meters, fume hood testing devices, thermometers, temperature humidity recorders, mechanical volumetric devices, oven vacuum gauges, and sampling interface flow controllers. procedures for ensuring the accuracy of the test equipment are summarized in the following sections. Additional information can be found in ATL Certification of Test Equipment SOP, #34, and Refrigerator and Freezer Temperature Monitoring SOP #19.

9.4.1 Analytical Balances and Weight Sets

The analytical balances are certified and serviced once a year by an independent balance maintenance company. A sticker is put on the side of balance to indicate the date of certification and the company performing the certification. The certificates are maintained in the Quality Assurance (QA) Department. The certificate must indicate that the reference standards are traceable to NIST standards and indicate the tolerances of the balance.

In addition, each time a balance is used, it is first checked with Class 1 weights. The weights used must bracket the final amount being weighed. The result must be within acceptance criteria. If the acceptance criteria are not met, a Corrective Action Request (CAR) form is initiated and given to the QA Department. The balance and/or weight set may require servicing to correct the problem.

Annually, all Class 1 weight sets are sent out for service and calibration against NIST-certified standards by an independent calibration company or the manufacturer. The certificate of calibration is maintained in the QA Department. The weights are kept in the manufacturer package that indicates the certification expiration date for the weight set. A sticker is put on the outside of the box to indicate the date of certification and the company performing the certification.

9.4.2 Pressure Gauges

Pressure gauges are used to verify sample receipt pressures and for gaseous standard preparation. The measurement of pressure on the gauges used to pressurize canisters is relative. The readings are used to assess the initial canister receipt vacuum/pressure and then pressurize the canister to a known pressure. The receiving personnel compare the final vacuum/pressure recorded on the Chainof-Custody Record and/or sample tags by the personnel with the receipt field vacuum/pressure. If there is a discrepancy of more than 5"Hg/5psi a Sample Discrepancy Report is initiated and the client is notified.

In addition, the pressure gauges installed on the pressurization manifolds are sent off-site for re-calibration and NIST certification every 6 months or as-needed. The certificates are kept on file in the QA Department.

9.4.3 Fume Hood Testing Device

Quarterly, the Velocicheck Portable Air Velocity Meter is used by a member of the Safety Committee to check fume hood velocities. Velocities are checked in various quadrants of the hood at both full open and half-open sash levels. Results of this check must be within specified limits and are recorded in the Fume Hood Evaluation Logbook. If results are outside of these limits, the fume hood must be taken out of service until the problem is corrected.

Annually, the Velocicheck Portable Air Velocity Meter is returned to the manufacturer to be calibrated against NIST-traceable standards. The Certificates of Calibration are maintained in the QA Department.

9.4.4 Thermometers

9.4.4.1 Reference Thermometers

ATL has NIST-traceable digital thermometers, which are used by the Technical Services group as reference devices. The thermometers are sent to the manufacturer for calibration and certification every year. A label indicating the date of calibration, the due date for the next calibration, and the name of the company performing the certification is placed on the back of the thermometer itself. The reference thermometers are kept, along with certificates of calibration, in the QA Department.

9.4.4.2 Ref/Freezer and Receiving Liquidfilled Thermometers

Thermometers used to record the temperature refrigerator/freezers as well Temperature Blanks (received with samples shipped on ice), are re-certified every year by the Technical Services group using the NISTtraceable digital thermometer as reference. The certification test is performed by comparison to the NIST-traceable digital thermometer. Both thermometers (working and reference) are placed in a Dual Well Dry Calibrator 9009 Block (Model Scientific). This instrument allows accuracy checks at both low and high temperatures. Alternatively, the certification test may be performed by placing the thermometers in a medium to large-sized beaker filled with water (or Methanol for freezer thermometers). The beakers are placed into a refrigerator (or freezer) for approximately one hour. The beakers are then removed from refrigerator/freezer and the thermometer readings are compared to the NIST-traceable digital thermometer which is also submersed in the liquid.

The temperature range tested must correspond to the temperature range that the thermometer is used to measure (i.e. approximately $4\pm2^{\circ}\text{C}$ for Refrigerator and Receiving thermometers and $\leq -10^{\circ}$ C \pm 5°C for Freezer thermometers). The results of this test are recorded in the Thermometer Check logbook. The difference between the temperatures of the working thermometers and the reference thermometer should be within the specified accuracy limits. Any thermometer that fails this certification test is discarded and new replacements are purchased as needed.

The manufacturer provides the newly purchased thermometers with a calibration certificate. The Technical Services group checks the new thermometers for defects (i.e., air bubbles present in red liquid column) before placing the new thermometers into use. A table containing the exact location, serial numbers, calibration and re-calibration due dates of each thermometer is kept in the Thermometer check Logbook.

9.4.4.3 Oven and IS Station Thermometers

Temperature controllers used in the Canister Cleaning and Tube Preparation areas are verified on a yearly basis by the Technical Services group to ensure that the proper temperature range is being achieved. The test is performed using the NIST-traceable digital thermometer as a reference. The test consists of comparing the temperatures displayed by the ovens temperature controllers versus the temperature measured by the reference thermometer.

The temperature range tested must correspond to the temperature range the thermometer is used to measure (i.e. approximately 65 - 125 °C for Can Cleaning ovens). The temperature readings are recorded in the appropriate logbook, which is kept in the QA Department.

The accuracy limits used to compare the two readings are \pm 5°C.

If the readings are outside of acceptance limits, a correction factor may be applied to the temperature readings and maintenance or replacement of the controller may be necessary.

Temperature control used for desorption at the Internal Standard (IS) Loading Station in the main lab (used for VOST and TO-17 analysis) is verified for accuracy on a daily basis using a NIST-traceable digital thermometer as a reference.

The temperature range tested must correspond to the temperature range the thermometer is used to measure (i.e. approximately 180 °C for the IS station). The temperature readings are recorded in the instrument logbook. The acceptance limits are \pm 10°C.

If the readings are outside of acceptance limits, a correction factor may be applied to the temperature readings and maintenance or replacement of the thermometer may be necessary.

9.4.4.4 Non-Contact Thermometers

Non-contact thermometers are used to verify the temperature of the Desorption plate used in the analysis of VOST and TO-17 samples as well as to take the temperature of chilled samples which arrive without a Temperature Blank. These thermometers are sent off-site on a yearly basis to be certified against NIST-traceable standards. Certificates of calibration are maintained in the QA Department.

9.4.5 Temperature/Humidity Recorders

A Temperature/Humidity Recorder is used to verify that conditions required for PM10/TSP analyses of filters have been met in the Desicator unit. The required conditions are a temperature of 59 to $86^{\circ}F + 5^{\circ}F$ and humidity at 20 to $45^{\circ}\%RH \pm 5\%$ RH ($\leq 50\%$ RH $\pm 5\%$

RH for TSP) over a 24 hour period. These conditions are graphed, and every 7 days a replacement graph card is placed into the recorder by designated personnel. The date range recorded, along with the analyst's initials, is noted on the back of the graph and filed in a folder next to the instrument. The recorder is sent off-site for re-calibration and certification after approximately every 6 months of use. The Certificates of calibration are kept on file in the QA Department.

The Refrigerator used for the storage of VOST samples (Refrigerator #4) uses a Temperature Recorder in order to verify required temperature has been maintained over weekends and/or holidays when the regular twice daily temperature checks are not performed (see ATL SOP #19 Refrigerator/Freezer Temperature Monitoring and Documentation). The temperature is graphed, and every 7 days a replacement graph card is placed into the recorder by designated personnel. The date range recorded, along with the analyst's initials, is noted on the back of the graph and filed in a folder next to the instrument. The recorder is sent off-site for re-calibration and certification after approximately every 6 months of use. The Certificates of calibration are kept on file in the QA Department.

9.4.6 Flow Meters

Flow meters are used in the Laboratory to check the flow rates for VOST/TO-17 and other analyses, and in connection with sorbent tube preparation. Canister Cleaning also uses flow meters to calibrate flow controllers. These instruments are sent off-site to be recertified annually against NIST-traceable standards. The certificates of calibration are kept in the QA Department.

9.4.7 Mass Flow Controllers

The Mass Flow Controllers on the sampling interfaces are used as part of the Initial Calibration. Therefore, measurements made

using them are relative in nature. The samples are introduced through the very same process, therefore any potential bias is self-correcting. In addition, the accuracy of the Mass Flow Controllers is verified in four ways:

- 1) Each time the daily CCV is analyzed, the recoveries document the accuracy of the Mass Flow Controller with respect to the most recent instrument Calibration.
- 2) The linearity of the Calibration Curve demonstrates the accuracy of the Mass Flow Controller because the curve is developed using a mixture of syringe and Flow Controller standard loadings.
- 3) The accuracy of the Mass Flow Controller is verified through comparison of the new Calibration Curve with the previous Curve
- 4) In addition, the Mass Flow Controllers on the sampling interfaces are calibrated in house using a NIST certified flow meter before each Initial Calibration. They are only calibrated by the Technical Services group or a trained Scientist. This action is documented in the instrument logbook the day of the calibration which includes flow controller serial number, NIST flow meter expiration date, nominal value, actual value, verified/set by initials and date. The Technical Services group oversees the Mass Flow Controller certification program. The certificate of calibration for

the NIST flow meter is kept in the QA Department.

9.4.8 Mechanical Volumetric Devices

Mechanical volumetric devices such as solvent dispensers are verified for accuracy against a known volume approximately once per month and never less than four times per year.

9.4.9 Oven Vacuum Gauges

Each oven used by the Support Services Dept. to clean stainless steel canisters is equipped with a CONVECTRON vacuum gauge and controller. The accuracy of these gauges is checked approximately every 6 months or as needed.

A NIST certified CONVECTRON gauge and controller is mounted onto an empty port on the evacuation manifold by a member of the Support Services Dept. The controller readings are compared to the oven vacuum gauges and recorded into to the comment line of the oven logbook. The NIST gauge and the oven gauge should match within \pm 6 % (\pm 1.2 mTorr for a 20 mTorr reading) based on the manufacturer's accuracy limits. If the 2 gauges do not match, then the oven vacuum gauge controller is adjusted until the readings are the same, or the oven gauge is replaced/repaired.

Documentation of changes or repair is noted in the Canister Cleaning Maintenance Logbook. The NIST gauge is sent for off-site recertification annually.

10.0 PROFICIENCY TESTING PROGRAM

10.1 NELAP PT SAMPLE PROGRAM

Proficiency testing (PT) samples are used to measure analytical accuracy, precision, and report completeness. To be accredited under NELAP, the laboratory contracts with an outside approved PT sample provider in each field of testing. Testing is limited by availability of samples that meet NELAP criteria (noted below). The provider must be a NIST accredited PT provider. It may be necessary to participate in more than one proficiency testing program to be evaluated for multiple interdependent analyte groups. Performance samples are processed through the laboratory in the same manner as project samples. In each calendar year, the certified lab will complete at least two separate proficiency testing samples for each analyte or interdependent analyte group. The following policies apply to laboratory PT sample analysis and reporting:

- The samples shall be analyzed and reported to the PT provider within 45 calendar days of receipt or the specific deadline specified by the PT provider.
- The laboratory must follow the PT providers instructions for preparing the PT sample.
- The laboratory management and bench chemist ensure that the PT samples are analyzed and reported in the same fashion as field samples using the same staff, equipment, and methods.
- The PT sample cannot undergo duplicate or replicate analyses that would not ordinarily be performed on field samples.
 The PT sample result cannot be derived from averaging the results of multiple analyses unless specifically called for in the reference method.

- The PT sample can only be analyzed on equipment leased or owned by the company and handled only by bona fide employees of the company.
- The analysis of PT samples by temporary or contract employees is explicitly forbidden.
- The laboratory shall not subcontract any PT sample or portion.
- The laboratory shall not knowingly receive any PT sample or portion from another lab.
- The laboratory shall not communicate in any fashion with another laboratory concerning the PT sample or results.
- The laboratory shall not attempt to obtain the PT sample result prior to reporting.
- The PT sample reporting forms provided by the sample provider will be maintained in the laboratory's record system.
- The laboratory shall maintain copies of all written, printed and electronic records relating the analysis or reporting of the PT sample for a period of 5 years.
- A CAR form will be generated any time an analyte result fails the proficiency testing assessment. A copy of the PT results is sent to the NELAP accrediting agency and associated corrective action summary will be sent upon request.
- The lab authorizes provider to release any PT assessment information to the accrediting agency.
- The QA Manager must sign the PT results form and by so doing, attests that the sample was analyzed and reported in the same fashion as a field sample and

followed the PT provider instructions for preparation.

- The lab must notify its primary accrediting agency and any other agencies under reciprocity that it has enrolled with a particular PT provider.
- The lab must notify its primary accrediting agency and any other agencies under reciprocity in the event it wishes to change PT providers.
- For each analyte or interdependent analyte group for which proficiency is not available, the certified lab will establish, maintain and document the accuracy and reliability of its procedures through a system of internal quality management.

10.2 EXTERNAL (NON-NELAP) PT SAMPLES

Occasionally proficiency testing samples are submitted along with field samples by private clients. The lab processes and reports the samples in the same fashion as field samples. When the client notifies the laboratory that one or more analytes appear to have failed, the report is processed through the normal Client Inquiry Corrective Action Process. The QA Manager will carry out an assessment and investigation into the circumstances surrounding the proficiency results including aspects relating to how the client prepared the sample for submission. The outcome of the assessment will be documented as per (Section 3.3.2) and maintained on file for a period of 5 years. Results of any failed external PT samples are summarized in the quarterly and of annually 'Status QA Report' management.

11.0 MANAGEMENT OF COMPUTER AND SOFTWARE SYSTEMS

Data are electronically captured from virtually all analytical instruments used by Air Toxics Limited. A network of computers and servers is used for the acquisition, processing, manipulation, recording, storage, and retrieval of test data. The laboratory uses a variety of both commercial as well as proprietary software applications to acquire, process, and report sample results. This network of computers is also used to receive and process customer information regarding field activities, sample disposition, and quality assurance objectives. Quality systems relating to the management of the computers and software are designed to incorporate the standards established in the EPA Document "2185-Good Automated Laboratory Practices (1995)" wherever possible given the size and resources available in the laboratory and IT groups.

11.1 SECURITY

The systems of Air Toxics Ltd. are protected from unauthorized access through the use of both physical and programmatic security measures. All of the laboratory servers are housed in a locked office, which maintains favorable environmental conditions to allow for optimal server performance. Access to the laboratory's networks is granted by the Systems Administrator or IT Manager. Network access is tightly controlled for the entire company. Users maintain individual network accounts and are allowed to access specific areas of the network based on the privileges assigned to them. A user is granted access to only those areas needed to fulfill his/her job function. All software used to reduce sample data or generate sample reports is password protected; users are granted rights to these systems based on a read/write/none privilege system.

11.2 BACK UP AND STORAGE OF DATA

All data systems are backed up on a daily, weekly, and monthly basis using a modified grandfather-father-son (GFS) rotation Specifically, these back ups are protocol. conducted on the servers responsible for all laboratory production data files and databases (i.e., Client Services files, analytical data, audit trails, quality assurance documents, etc.). A daily incremental back up is scheduled to run each night Monday through Saturday. The daily incremental back up is limited to files modified the same day. On Sunday, a weekly full back up of all files on each server is completed. At the end of each month, a full back up of each data system is conducted. This monthly back up tape is then placed in permanent storage. The permanent historical back-up tapes are stored in a fireproof safe in the secured server office. Data is not removed from the server until at least three permanent monthly back-up tapes have been created. This ensures that no archived data will be lost due to corruption of the magnetic tape. A more comprehensive description of the electronic data archiving system can be found in ATL SOP #55, Electronic Archival of GC/MS Analytical Instrument Data.

11.3 SOFTWARE AND ELECTRONIC DATA VALIDATION

The IT department is responsible for the testing and verification of all internally developed software applications. This includes testing during the software development, testing of the first pre-release version (alpha testing), and testing the release version in a closely monitored production environment. Findings discovered during the alpha and beta tests are documented and software fixes are applied as warranted. All custom software applications are tested prior to their release to the production laboratory.

There are three stages of testing and implementation of custom software modules by the IT department.

11.3.1 Stage I – Alpha Testing

Stage I testing is conducted for all ATLAS modules developed at Air Toxics Ltd. The Stage I testing is performed by two members of the ATLAS development team, typically the lead programmer and a representative enduser. The lead programmer is responsible for preliminary testing of the application and fixing errors, "bugs," within the program. The other evaluator acts as the user, testing the functions and features of the ATLAS module. The alpha test performed by the representative end user is conducted at a production workstation, which maintains the typical ATL configuration and connects with the ATLAS database through the laboratory network. This Stage I testing is conducted off-line, as the module is not used to perform any real-time production work. During the testing, the alpha tester identifies errors within the program and reports them to the lead programmer via the ATLAS Bug Report form. Examples include: finding areas where the module does not perform the task as per the specifications or an error message is displayed on the screen when working with the module.

The lead programmer evaluates the errors that are reported on the Bug Report form and fixes all identified problems within the ATLAS module, documenting the status of the repair on the Bug Report form (Exhibit 11.1).

Once all main functions have been tested and the alpha tester can no longer identify significant bugs within the program, the alpha testing is concluded. The alpha test results are documented in the completed ATLAS Bug Report forms, which are maintained as a permanent record in the IT Manager's office.

11.3.2 Stage II – Beta Testing

The Stage II testing is conducted after completion of the alpha test. This testing is performed by select on-line end-users. End-

user participants in the beta test are chosen by their department manager, in an effort to have an accurate representation of the end-users that will be using the ATLAS module. The beta test participants perform their job duties using the ATLAS module. Both the lead programmer and the IT manager are available during the beta test.

The beta testers are trained how to use the ATLAS module on-line. The lead programmer, IT Manager, and the department supervisors all participate in the training of the beta testers. The beta testers perform their production duties on the ATLAS module and check all output from the module for accuracy. A summary of the beta test may be documented (Exhibit 11.2).

The beta testers identify any errors or deficiencies within the application and notify the lead programmer immediately upon finding a bug. In addition, the beta testers submit an ATLAS Bug Report form to the programmer.

The lead programmer evaluates the errors that are reported on the Bug Report form and fixes all identified problems within the ATLAS module, documenting the status of the repair on the Bug Report form.

The new version of the ATLAS module is uploaded onto the computer for the beta tester. The testing is resumed at the beginning of the task from which errors were identified in the software application. The tester verifies the fixes for reported bugs and documents this verification on the Bug Report form.

After the occurrence of bugs has been mitigated, the beta testing is concluded. The beta test results are documented in the completed ATLAS Bug Report forms, which are maintained as a permanent record in the IT Manager's office.

11.3.3 Stage III - Implementation

Stage III implementation is initiated after completion of the beta test. All end-users are given access to on-line module. Training for end-users is conducted by the members of the ATLAS development team and the department supervisors. Users are trained individually by the department supervisors and/or a member of the ATLAS development team.

If any bug occurs on-line, a member of the IT team is contacted and the problem assessed to determine if it is a bug or a training issue. A confirmed bug is documented on an ATLAS Bug Report form. Any problem that affects data integrity is documented separately. This type of problem is typically documented by the IT Manager, however other members of the development team can assume this responsibility. This documentation is kept as a permanent record in the ATLAS project documentation binder in the IT Manager's office.

The lead programmer evaluates any error reported on the Bug Report form and fixes the program within the ATLAS module, documenting the status of the repair on the Bug Report form. The new version of the ATLAS module is uploaded to all PCs running

the application. In most cases, an automatic update program loads the new version of the application for all users when they re-enter the program. Production work continues with the new version of the module.

11.3.4 Commercial Data Validation

Any commercial software application used for data reduction is verified for adequate performance before release to the production laboratory. Reports created by laboratory's LIMS are reviewed for accuracy by the analyst responsible for creating the deliverables and a team scientist. entered results are checked for accuracy by the analyst responsible for this data entry. Peer review is also conducted on these reports. Deliverables created using the LIMS are spotchecked for accuracy. Documentation of these reviews is maintained as a permanent record in the Work Order folder. The various testing stages and process for implementation of internally developed software are described in ATL SOP # 47, Implementation and Testing of Modules, and ATL SOP Preparation Of Hardcopy Analytical Reports Using Automated Data Transfer.

Exhibit 11.1. ATLAS Bug Report

Mod	lule: Accounting Quote Methods Quotes Project Profiles Contacts Other:				Shipments System Administration Work Order Editor (ATLAS98) Method Editor (ATLAS98) Compound Editor (ATLAS98) SuperCATS Accounts	
Des	cription of Problem:					
						_
				·		_
		-				_
						_
Rep	orted By:		Date:_			
	us:		Pending		☐ Could not duplicate	
	Other:					
						_
				<u></u>		_
	**************************************					-
				······································		_
Vor	ified as Fixed \square Dr				Data	

Exhibit 11.2. ATLAS Beta Testing Summary Form

Name of Tester:	Date:			
Module Tested:				
Summary of Beta Test (include brief description	of criteria used, etc.):			
Did the module pass all tests conducted?	□ Yes □ No			
If no, please provide comments below:				
	· · · · · · · · · · · · · · · · · · ·			
Signature of Tester:				
Module approved for release to production group	? □ Yes □ No			
Signature of IT Manager:	Date:			



12.0 CONTROL OF PURCHASED ITEMS AND EXTERNAL SERVICES

The primary materials procured by the laboratory are computer hardware, analytical software, standard office software, analytical instrumentation, certified standards, carrier gases and cryogens, miscellaneous laboratory supplies, NIST-traceable re-certifications, disposable sampling media (e.g., Tedlar bags), and service contracts.

Control of the purchase of these items and services is maintained using a standard purchase order system that includes the following:

- A purchase request that is approved by a director or manager.
- An assigned purchase order (PO) number that is logged along with the date, vendor, and requester.
- A requirement that upon receipt or delivery of services, the product is inspected by the purchasing agent, and compared to the packing slip and/or request for services.
- Each PO is matched with invoices prior to payment to insure that purchased items or services were delivered as expected.

Critical vendors (those for whom a failure or lack of supply would cause irreparable damage to the laboratory operations) are selected on the basis of either being the sole supplier of an item or as a result of reliable service over a several year period. A table of current critical suppliers is presented in Exhibit 12.1.

When reagents are purchased in bulk volume for laboratory use, each lot is certified for cleanliness prior to use. A laboratory blank is prepared using the analytical reference method. The concentration of target species present must be less than the laboratory reporting limit for the lot to be certified clean

and approved for use. Once a lot is approved, the vendor will set aside stock sufficient for several months for ATL use. The certificates of analysis are kept on file in the main analytical laboratory and each incoming shipment is monitored by the extractions area staff to verify that only certified lot numbers are used in the processes.

Solvent **Certification Method** Methylene Chloride SW8270C

Hexane TO-4

Water SW8260B/SW8270C

Each reagent is labeled with the date of receipt, date opened, and date of expiration. In the case of Methylene Chloride and Hexane, a 100 mL aliquot of the solvent is evaporated to 1 mL and analyzed via method SW8270C or TO-4. In the case of reagent water, a reagent blank is prepared and analyzed via methods SW8260B (5 mL) and SW8270C (1 L). Only reagent grade water is used in the laboratory and is purchased in bulk lots as HPLC grade.

Cylinder gasses used as diluent gas for sample analyses are also certified for cleanliness. Each tank of UHP Helium or Nitrogen is tested by filling a clean lab blank canister followed by analysis via EPA method TO-14. All target species must be present at less than the reporting limit in order for the cylinder to be certified clean. Results of the analysis are posted on each cylinder and stored in designated logbooks.

NIST traceable standards are re-certified upon receipt. The standard is analyzed under the appropriate method and compared to the existing inventory of standards. response of any target species is not within acceptance limits for a second source standard. the standard is considered to have failed. The vendor is then contacted to discuss the discrepancy and arrangements made to replace the standard.

Exhibit 12.1 Critical Suppliers

VENDOR	JUSTIFICATION
Scott Specialty Gases	Sole supplier/Reliable service over several years
Airgas	Reliable service over several years
Spectra Gases Inc.	Reliable service
SKC West Inc.	Sole supplier
Chromatography Research Supplies, Inc.	Sole supplier
Scientific Instrument Services	Reliable service over several years
Hamilton	Reliable service over several years
Quantum Analytics, Inc.	Reliable service over several years
R & D Glassware	Reliable service over several years
Aldrich Chemical Company	NIST Certified
Supelco	Reliable service over several years
Valco	Sole Supplier
VWR	Reliable service over several years
Control Company	Reliable service
Agilent Technologies	Reliable service
Davis Calibration Laboratory	Reliable service
Dickson Calibration Services	Reliable service
Sierra Instruments Inc.	Reliable service
Millipore/Mykrolis	Reliable service over several years
Leco Corporation	Sole Supplier

13.0 PROJECT MANAGEMENT SYSTEM

Any quality system, no matter how elaborate, and no matter how well documented, will fail unless the customer expectation is effectively communicated. The ATL Project Management System describes the critical pathways necessary to adequately ensure that the customer expectation is reviewed, committed to by top management, documented and communicated to the laboratory prior to sample delivery. System elements include:

- ✓ Review of project specific documents
- ✓ Negotiations and variance requests
- ✓ Documentation of project requirements
- ✓ Documenting client discussions
- ✓ Project briefings
- ✓ Scheduling sampling media
- ✓ Tracking sample analysis and reporting
- ✓ Project follow-up

The Project Management System is defined in the ATL SOP #1. Following are brief descriptions of the elements comprising project management systems.

13.1 REVIEW OF PROJECT SPECIFIC DOCUMENTS

Clients document project requirements in requests for proposals, work plans, SOWs, or QAPPs. No matter how the details are documented, the project requirements must be reviewed to ensure that the laboratory has sufficient staff, equipment, and capacity to meet project needs. Any document received from a client

containing either contractual language, description of work, OA/OC criteria. and/or deliverable requirements different from our Standard Terms and Conditions will be processed as a proposal. When a proposal is received, an electronic Proposal File is started and routed through the Proposal Tracking System. Account Managers are provided with technical support by the designated project chemist who reviews all relevant sections of the proposals. Each department's team leader is consulted for further information when needed.

Items to be reviewed include:

- Requested methods
- Adequate and documented training of appropriate personnel including Demonstration of Capability
- Adequate instrumentation
- Target compound lists and reporting limits
- Quantity of samples
- Requested media and degree of preparation
- Media preservation requirements
- Holding time requirements
- Project specific QC
- Deliverable requirements
- TAT requirements
- Insurance requirements
- Special billing and payment terms
- Data storage requirements
- Client financial status

The following flow chart (Exhibit 13.1) depicts how the proposal is distributed through the review process. Project requirements are received and logged into

the tracking system database. Each proposal is given a unique identifier at that time. The Project Chemist reviews the document to determine whether or not the proposal contains methods, target compounds, or production demands requiring additional review. The Project Chemist reviews the document for special project requirements in the areas of:

- Quality assurance criteria
- Reporting criteria
- Electronic deliverable criteria
- Unusual TATs
- Unusual volume of samples
- Unusual compounds

If the Project Chemist has any questions, which may affect production, then the Account Manager is notified and the proposal is sent to the Operations Director to determine the implications to the laboratory production throughput. With Operations approval, the proposal may be sent to the Technical Director or designee for review and comment. If the proposal does not require method development, and has unusual deliverables then it is sent to the IT Director or designee to verify ATL is able to meet client deliverable demands. The reviewer may suggest pricing adjustments according to the client specific format difficulties. If the proposal does not require review by the IT Manager, it is returned to the Project Chemist. A variance table to be included with the proposal may be created by the Project Chemist. The variance table may undergo revision as per the result of discussion with the client.

The Contract specifications are reviewed by the Contract Administrator to evaluate the terms and conditions of the proposal, including the following areas:

- Retention;
- Penalties;
- Sales and Use Tax Requirements;
- Insurance Requirements;
- Data Storage Requirements; and
- Payments Terms.

The Contract Administrator then reviews the proposal to determine the amount of credit to be established for the client. The amount will be based upon credit status (Dunn & Bradstreet) report, the amount of the contract and past payment history with ATL.

Each of these steps is documented on the Bid/Proposal/Contract Review sheet (Exhibit 13.2). The review and comment activity is documented by signature and date on the Bid/Proposal/Contract Review sheet.

Following review and comments, the proposal is returned to the Account Manager for final review and pricing. The Client Service Representative is consulted and the proposal is generated. Then the proposal is entered into the ATLAS Ouotes module for reference. If the bidding process is successful, the proposal documents continue on through the Client Services Representative to be set up as an Before samples are active project. received and the analyses are performed, a signed copy of the variance table must be scanned to the network. The Project Chemist is brought back as an active team member at this time if negotiations are The Client Services necessary. Representative will complete the project profile, while the Project Chemist creates a project requirement table that includes the approved variance table in the laboratory sample tracking database. An example of

project profile appears in Section 3, Exhibit 3.1.

Before the final report is released a contract must be signed and filed. Once a contract is received, it is documented in the contract log. The contract is then

reviewed to compare the pricing and language to the proposal. If there are exceptions made to the contract, these are noted and negotiated with the client. Once pricing and terms have been agreed upon, the contract is then executed and copies sent back to the client.

Exhibit 13.1 Proposal Review Flow Chart

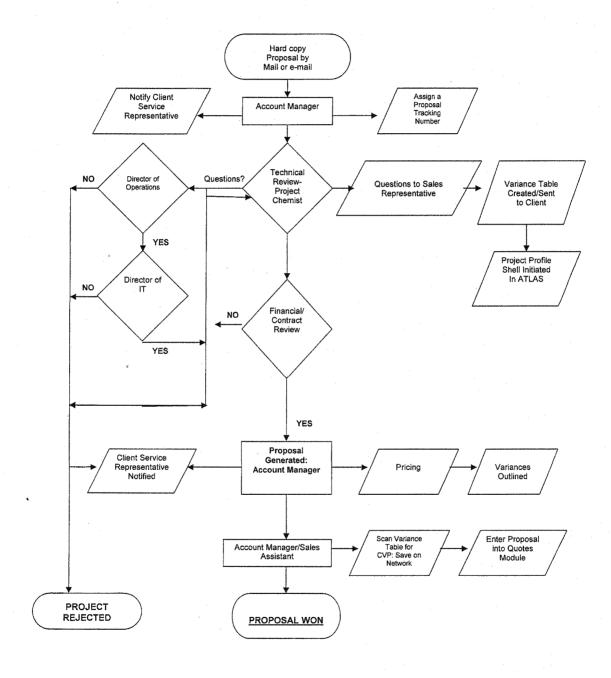


Exhibit 13.1 Proposal Review Flow Chart (Continued)

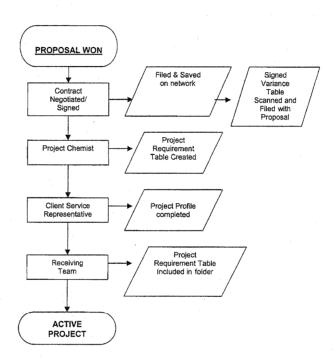


Exhibit 13.2 BID/Proposal/Contract Review Form

CLIENT	DUE DATE:
PROFILE#	DATE RECD:
PROPOSAL TRACKING#:	AM:
BID PROPOSAL (REVIEW)	
PROJECT NAME: RFP#	•
1. ACCOUNT MANAGER REVIEW – FEASIBILITY QAPP? Yes No	DATE DUE:
SOW? Yes No	Yes No
Reviewed by: Date	
2. TECHNICAL REVIEW	DATE DUE:
A. REVIEWER #1	DATE DOLL
Variance Table? Yes No Notes Page? Yes No Special Compounds? Yes No (If ye required)	s, a special compound request form is
Reviewed by: Date	ə:

B. REVIEWER #2		
Variance Table? Yes No Notes Page? Yes No Special Compounds? Yes required) Comments:	No (If yes, a special comp	oound request form is
Reviewed by: C. LAB MANAGER (Optional)	Date:	
Special Technical Requirements Comments:	Yes No	
Designed by	Data	

DELIVERABLES - IT MANAGER /TEA	AM E		DATE DUE:	
Client Specific Format: Yes No eCVP Yes No Comments:	Format?			
Reviewed by:	Date:			
FINANCIAL REVIEW		DATE DUE:		
A. CONTRACT REVIEW - FINANCE				
Contract Provided? Yes Exceptions Taken? Yes Retention Exceptions? Yes Penalty Exceptions? Yes Insurance Exceptions? Yes	No No No No No			
Data Storage Exceptions: Yes Payment Terms Exceptions: Yes Comments:	No No			***************************************
Reviewed by:	Date:			-

	B. FINANCIAL BACKING REVIEW		
	Credit information on file Yes No Terms & Conditions on file? Yes Dunn & Bradstreet #: Rating: Comments:	No No	- -
	Reviewed by:		
i,	CSR REVIEW		DATE DUE:
	Variance Table Required? Yes started) Comments:	No (If	yes, ensure a project profile has been
	Reviewed by:	_Date:	
š.	RETURN TO AM TO FINALIZE PROPOSAL		DATE DUE:
	Reviewed by:	_ Date:	
	SIGNATURE		DATE DUE:
	Reviewed by:	_ Date:	, and and man, man and analyst talked allow place talks that there are near result
ì.	RETURN TO AM TO FILE		DATE COMPLETED:
	Variance Table Signed? Yes Proposal saved to Network? Yes Additional Information:	No No	

13.2 Negotiations and Variance Requests

When the Project Chemist notes differences between the project request and laboratory standard protocol, the laboratory may request a variance from the Ideally, variance requests requirements. occur during the proposal stage, but regarding sufficient details project requirements are sometimes not known until sampling is about to begin. With the assistance of the QA team the Project Chemist notes all discrepancies in a variance table that is submitted to the Account Manager. The assigned Account Manager communicates the discrepancies to the client by faxing the variance table. Variance requests are most often handled directly between these parties. occasion, a conference call may be held with agency representative or additional project and laboratory staff present. It is the responsibility of the Account Manager to coordinate the meeting.

Once an agreement has been reached, the Project Chemist and the QA team will finalize the understandings concerning the discrepancies in a final variance table, which is used for sample login and analysis. The variance table is given a unique identifier that ties it to the unique identifier assigned to the proposal. The table is stored on a network-shared drive in read-only format until sample log in occurs.

13.3 DOCUMENTATION OF PROJECT REQUIREMENTS

The Client Services Representative becomes the primary client contact following the project award. The Client Services representative will verify at that time that the client has been provided most recent version of the project plans. Any outstanding issues are discussed by phone and documented in the customer contact database. At this time the project profile is updated to include:

- Shipping information
- Report To and Bill To information
- Pricing
- Reference method
- Requested media and degree of preparation
- Target compound list
- Special QA requirements
- Calibration criteria (if different from SOP)
- Deliverable requirements
- Variance table by reference
- Any special instructions
- Subcontracting (when relevant)

The project profiles are secured with read only privileges for all staff except the Client Services Representatives, Account Managers, the Project Chemist and members of the QA team.

13.4 DOCUMENTING CLIENT DISCUSSIONS

Once a project has been awarded, the majority of contacts occur via phone and are documented in the Client Services software. The software can track contacts by client name, project name, or date. Client Services team members may sort the database for summaries of calls made on the basis of specific clients or subcontracting entities.

13.5 PROJECT BRIEFINGS

Client Services Representatives hold frequent briefings to discuss the recent

client calls. The entire team is kept informed of the status of in-house projects. Potential scheduling problems or other matters affecting sample analysis and reporting are discussed.

13.6 SCHEDULING SAMPLING MEDIA

The ATL Client Services Representatives work closely with clients to ensure that media is delivered according to project schedule. Shipments for on-going projects are scheduled well in advance. The Client Services Representatives are responsible for processing shipping requests through the Client Services database. This is the same database used to log phone contacts. Each shipping request is given a unique identifier. Both laboratory staff and shipping staff monitor the database throughout the day for posted shipping requests.

Some media types (e.g., DNPH solutions, PUF/XAD cartridges, etc.) take time to prepare. Careful planning and scheduling on the part of both the project managers and the clients is necessary. Clients are encouraged to provide as much lead-time as possible.

13.7 TRACKING SAMPLE ANALYSIS AND REPORTING

Client Services Representatives monitor the progress of samples from the time they arrive at the laboratory until the final report has been delivered. The sample tracking database is reviewed daily and provides the Client Services Representatives with up-to-date information on sample status. Daily contact with the laboratory team and task leaders is necessary when the rate of the production is less than desirable in any area. It is the responsibility of the Client Services Representative to inform the client in the event that delays in analysis and reporting are anticipated.

13.8 PROJECT FOLLOW-UP

All contact with the client following reporting is handled by the Client Services Representatives. This includes requests for re-issues, perceived problems with further clarification. discussions are documented in the Client Services database. When a client desires a modification to a completed report, the Client Services Representative reviews the request. Commonly the laboratory team or task leader responsible for the work or the OA Manager may be consulted. If the request is considered to be legitimate, the Client Services Representative initiates a request for report re-issue (per ATL SOP #68) in the Sample Tracking database. The database tracks the re-issue status to make sure that the report is fixed and sent to the client. The reason for the re-issue is documented on the report cover along with report re-issue date. It is the responsibility of the Client Services Representative to ensure that the correct reason and supporting documentation is provided in the report folder.



14.0 DATA INTEGRITY PROCEDURES

14.1 Training

Data integrity is the cornerstone of Air Toxics Limited. Our Mission Statement and Value Statement define the goals and values which produce data of known and defensible quality. Everything from data reporting programs, employee retention programs to customer service programs evolve around maintaining our core values above all else.

ATL Mission Statement

- To deliver high quality cost effective environmental analytical services
- That are profitable, on time and meet/exceed the expectations of our customers

How we go about accomplishing this mission is governed by our standards of conduct; the ability to discern right from wrong and proper from improper and the commitment to always do what is right, good and proper.

"We seek success in what we do, but not at the expense of integrity. Integrity is essential to establishing and maintaining the trust that allows us to work as a team and to foster confidence in our customers and co-workers. Without integrity there can be no trust."

Without integrity, our customers would not trust us to get the job done and would not hire us. Employees would not trust management to set realistic goals and provide adequate resources. Without integrity, management would not trust employees with the ultimate form of empowerment.... direct responsibility for customer satisfaction.

All new employees are provided with introductory training which consists of:

Ethics Training I

The ATL Value Statement
The ATL Mission Statement
Definition of Standards of Conduct and Ethics
ATL Strategic Goals
Importance of Trust
Definition of Integrity
Four basic enemies of integrity with workplace
examples
Criminal ramifications for misconduct
Benefits of Integrity Training
Role of the Employee (reporting systems)
Role of Management (promote core values)
Where to find ATL Codes of Conduct
Employee Handbook (employment standards)
Quality Manual (quality standards)

SOP's (technical standards)
Chemical Hygiene Plan (safety standards)

vearly refresher course is provide

A yearly refresher course is provided which reviews the basic information of Ethics Training I but then goes on to define the critical steps in making ethical decisions:

Ethics Training II

- 1. Review relevant values and standards, devise a plan
- 2. Question the plan, your motives and any consequences
- 3. Resolve to address any ethical dilemmas by making touch choices.

Case studies are used in Ethics Training II to encourage work group participation in actual decision making scenarios. Following each training session, the employee is provided with a certificate for training and asked to sign the certificate indicating they have been informed of their obligations in the ATL integrity program and understand that legal ramifications may be imposed upon them for failure to comply.

14.2 Periodic monitoring

There are three parts to the ongoing periodic monitoring for inappropriate data manipulations following initial training. First, the IT department has removed access to adjustment of data acquisition and reporting computer clocks. Second, each new employee undergoes a training period in which all of their data is reviewed by a scientist or team leader until such time as basic knowledge and comprehension of method as well as data integrity procedures is demonstrated. A development plan is provided to each new employee by their Department Manager or Team Leader, which outlines goals. timelines and demonstration understanding. The measurement of success may include proficiency with a task as well as a verbal or written test demonstrating concept comprehension.

When the Department Manager and/or Team Leader determine the new employee is ready to produce data of defensible quality, he/she is asked to provide several representative data packages to the Quality Assurance Manager for thorough review. If the QA Manager agrees that defensible data was produced, the employee is released from the 100% review program.

The third and final part of the periodic monitoring program involves the use of proprietary in-house data validation software to review every data point generated and to alert the reviewer when manual integrations occur. The software is also programmed to report when more than three attempts for a daily CCV or tune check standard to pass have been attempted. (Validation software currently reviews all method TO-14A/15 results.

Further software development is scheduled in 2006 to bring the more methods on line.)

14.3 Mechanisms for Reporting Infractions

During Ethics Training I, the new employee is informed how to go about communicating any real or perceived infractions of the data integrity system. Open dialogue is encouraged between the employee and any member of management or senior scientists they feel most comfortable with. It is management's responsibility to relay the information to the Technical Director(s) and follow-up with an inquiry or corrective action. The employee who desires to remain anonymous is encouraged to report to the teams senior scientist as ombudsman. The scientist will meet separately management and the employee involved in order to ensure anonymity. An immediate inquiry by one of the Technical Directors will follow each and every reported incident. Documentation of the inquiry and subsequent disciplinary actions will be maintained by both the Technical Director and the Human Resource Manager.

DEFINITIONS AND TERMS

Accuracy: The degree of agreement between an observed value and an accepted reference value.

Analyte: The substance or thing for which a sample is analyzed to determine its presence or quantity.

Approved: The determination by any state for federal accrediting agency that a certified laboratory may analyze for an analyte under the specified method.

Assessment: The process of inspecting, testing and documenting findings for purposes of certification or to determine compliance.

Batch: A group of analytical samples (≤ 20) of the same matrix processed together including extraction, concentration and analysis using the same process, staff and reagents.

Bag: Means an inert air-sampling container consisting of inert polymeric material.

Canister: A stainless steel spherical air-sampling device consisting of summa polished or glass lined internal walls and a leak tight on/off valve.

Contamination: The effect caused by the introduction of the target analyte from an outside source into the test system.

Continuing Calibration Verification (CCV): A CCV is analyzed to verify instrument linearity with respect to the Initial Calibration. The CCV concentration may be identical to any given point contained within the initial calibration. A CCV is analyzed at the beginning of every analytical sequence (all methods) and then once every ten or twenty samples depending on the method (GC and LC, GC/MS excluded). GC and LC methods also include a CCV in every analytical sequence as an End Check.

Control Charts: These are statistical tools for monitoring the performance of a particular task on a continuing basis. The control chart is prepared for each test parameter after 20 determinations have been performed. The mean is plotted with the warning limits being $\pm 2s$ and the control limits being $\pm 3s$. (s – standard deviation)

Corrective Action: An action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Data Reduction: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Duplicate Samples: Samples collected for checking the preciseness of the sampling process. These samples are collected at the same time and from the same source as the study samples.

Equipment Blank: A sample that is known not to contain the target analyte that is used to check the cleanliness of sampling devices, collected in a sampling container from a clean sample collection device and retuned to the laboratory as a sample.

Field Blank: A sample that is known not to contain the target analyte and is used to check for analytical artifacts or contamination introduced by sampling and analytical procedures, carried to the

DEFINITIONS AND TERMS

sampling site, exposed to sampling conditions and returned to the laboratory and treated as an environmental sample.

Field Duplicate: Samples collected at the same time from the same source, but submitted and analyzed as separate samples.

Holding Time: The maximum time that a sample may be held prior to preparation or analysis.

Impinger: The glass vessel used to contain collection solution through which a stream of air is bubbled for sampling purposes.

Initial Calibration: Demonstration of a linear response to different concentrations of calibration standards within a defined range.

Initial Demonstration of Analytical Capability: The procedure described in the method 40 CFR Appendix A, used to determine a laboratory's accuracy and precision in applying an analytical method.

Instrument Blank: A sample that is known not to contain the target analyte, processed through the instrumental steps of the measurement process used to determine the absence of instrument contamination prior to analysis of field samples.

Instrument Detection Limit (IDL): It is the concentration of the analyte that produces a signal greater than five times the signal-to-ratio of the instrument.

Interference: The effect on the final result caused by the sample matrix.

Internal Standards (ISs): These are the measured amounts of certain compounds added after preparation or extraction of a sample.

Key Personnel: The laboratory director, technical director, quality assurance manager, and team leader, all of whom meet the requirements of the NELAP rule.

Laboratory Control Sample: An independent second source reference standard which goes through he same pretreatment and preparation procedures as the samples. It validates the accuracy of the initial calibration.

Laboratory Duplicates: Aliquots of the same sample that are prepared and analyzed at the same time.

Limit of Detection (LOD): an estimate of the minimum amount of a substance that an analytical process can reliable detect. An LOD is analyte and matrix-specific and may be laboratory dependent. The LOD may be determined by a Method Detection Limit study.

Limits of Quantitation (LOQ): the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. Generally, the LOQ is equal to the concentration of analyte(s) in the lowest point of a calibration (see Reporting Limit).

Matrix: The component or substrate (e.g., surface water, drinking water, air, liquid waste) which contains the analyte of interest.

DEFINITIONS AND TERMS

Matrix Spike: A sample prepared to determine the effect of the matrix on a method's recovery efficiency by adding a known amount of the target analyte to a specified amount of matrix sample for which an independent estimate of the target analyte concentration is available.

Matrix Spike Duplicate (MSD): Duplicates of the matrix spike sample.

Measurement Uncertainty: Measurement uncertainty is the estimation of potential errors in a measurement process and is expressed as $\pm 2X$ (s) of the historical mean of LCS recoveries.

Method Detection Limit: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero as determined from analysis of a sample containing the analyte in a given matrix (40 CFR Part 136, Appendix B, July 1995).

Practical Quantitation Limit (PQL): A synonym for the standard of lowest concentration contained in the Initial Calibration. It is the smallest concentration of the analyte that can be reported with a specific degree of confidence.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance or arrange, in either absolute or relative terms.

Preservation: The temperature control or the addition of a substance to maintain the chemical or biological integrity of the target analyte.

Proficiency Testing (PT) Assessment: The event including receiving, analyzing, and reporting of results from a set of samples that a proficiency testing provider sends to a laboratory for the laboratory to demonstrate compliance with the proficiency testing requirements.

Proficiency Test (PT) Sample: A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Quality Assurance: An integrated system of activities involving planning, quality control, reporting, and quality assessment and improvement to ensure that the product meets defined standards of quality with a stated level of confidence.

Quality Assurance Project Plan (QAPP): An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific data collection activity.

Reporting Limit: The smallest concentration of an analyte that can be measured with a stated probability of significance. All Initial Calibrations contain a standard at the Reporting Limit. The Reporting Limit is never less than the PQL.

Reporting Limit Verification: A re-quantification of the lowest concentration data point of an initial calibration to test the percent recovery of each component. Analyte recovery should be between 50-150% to verify detection limit accuracy.

DEFINITIONS AND TERMS

Selectivity: The capability of a method or instrument to respond to the target analyte in the presence of other substances or things.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels of a target analyte.

Standard Operating Procedures (SOP): A written document which details the steps of an operation, analysis or action whose techniques and procedures are thoroughly prescribed, and is accepted as the procedure for performing certain routine or repetitive tasks.

Surrogate: A substance which is unlikely to be found in the environment and which has properties that mimic the target analyte and that is added to a sample to check for analytical efficiency.

Target Analyte: The analyte that a test is designed to detect or quantify.

Technical Employee: A designated individual who performs the analytical method and associated techniques.

Trip Blank: A sample known not to contain the target analyte that is carried to the sampling site and transported to the laboratory for analysis without having been exposed to the sampling procedures.

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Quality Assurance Management Plan Chester LabNet

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This document supplants any previous versions.

Review History

Review Date	Changes	Author
2/7/06	Definition of "basic laboratory skills" added to Personnel qualifications, SOP formatting elements changed to reflect new NELAC compliant format, acceptance/rejection criteria for non-standard methods added, added section on documentation of customer inquiries/complaints, updated staff resumes, updated SOP listing, added 'failure to report' to the list of breaches of ethical behavior in Appendix D.	Sheri Heldstab
7/8/05	Expanded section 7.7, revised section 10.4 and 10.5 to reflect new internal audit process, updated staff references.	Sheri Heldstab
1/5/05	Added Appendix D.	Sheri Heldstab
10/21/04	Major updating to incorporate elements of NELAC/ORLAP requirements. Sections 3.3.2.1, 7.3 – 7.7, 8.2.4 -8.2.7, 8.3, 8.3.1 – 8.3.5, 8.4, 9.4, 10.6 – 10.8, 11.8 – 11.10 added.	Sheri Heldstab
1/23/04	Updated floor plan and Capital inventory table. Minor grammatical changes.	Sheri Heldstab
3/10/03	Changes in organization of Section 3. No changes to content, only to formatting.	Sheri Heldstab
12/10/2002	Minor changes to text, some text rearranged for clarity and conciseness.	Sheri Heldstab
11/8/2001	This document has been in existence since at least 1988. The number and types of revisions are unknown. As of this date, the document is being revised to standardize the formatting of Chester LabNet documents.	Sheri Heldstab
Unknown	No Changes. Date of origination.	C.R. Lytle

Author Signature _		
_		
Date		

Concurrences

The following approved signatories, by their signature, attest to having read and understood the most current version of the Quality Assurance Management Plan for Chester LabNet:

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1.0 Introduction

Chester LabNet specializes in Inorganic Air Quality Analysis of ambient air and source emissions, including analysis of PM10 and PM2.5 samples.

Quality Policy Statement: Our goal is to provide the most informed and accurate inorganic analysis of air quality samples available from a commercial laboratory. Chester LabNet's management is committed to good professional practice and to the quality of its environmental testing in servicing its clients. This document defines its policies and objectives for, and its commitment to, accepted laboratory practices and quality testing services. All personnel concerned with environmental testing and calibration activities within the laboratory are familiar with the quality documentation requirements and implement the policies and procedures in their work as attested to by their signatures on the concurrences page of this document. The laboratory and its management are committed to complying with all requirements of any accreditations, contracts and governmental mandates.

Chester LabNet is proud of having specialized in the inorganic analysis of ambient particulates and source emission samples since its inception (as NEA, Inc.) in the late 1970's. The laboratory as an organization, its management and its personnel are all committed to the production of the highest quality data achievable with current methodologies and instrumentation, as well being committed to complying with contractual and accreditation standards and requirements.

1.1 Quality Assurance Management

For any activity involving a service or the creation of an analytical result, quality may be defined as conformity to a given set of requirements. To ensure acceptable quality, three conditions must be met: (1) requirements and objectives must be clearly delineated before work begins; (2) the major steps in the production of the service or analytical result must have a component that allows for the control of quality, based on the end-result objectives; (3) the components of quality control must include control limits and corrective actions designed to both effectively monitor quality and modify procedures if quality is compromised.

Quality Assurance (QA) comprises the overall program elements designed to maintain any activity within the stated objectives. Examples of such program elements are: clearly stated precision and accuracy targets; written standard operating procedures for all laboratory and instrumental protocols; the selection of sample preparation and analytical methods that are most appropriate for the matrices and analytes to be encountered; etc.

Quality Control (QC) comprises the individual checks used to monitor laboratory procedures, the precision and accuracy statistical control limits for each individual check, and the specific corrective actions to be followed when QC results are outside control limits. An example of a QC element is the matrix spike. Good quality control would set the frequency of analysis, the particular QC statistic to be used (e.g. percent recovery), the control limit (based on published statistics for the particular analysis or on QC charts developed in house), and the corrective action for QC results that are out of control.

1.2 The Chester LabNet Quality Assurance Management Plan

The objective of the Chester LabNet QA Management Plan (QAMP) is to provide a unified approach to quality assurance that ensures the production of data that are scientifically sound, legally defensible, and of known and acceptable quality.

The QA Management Plan (QAMP) represents the Chester LabNet objectives, policies, organization, functional activities, and specific QC activities for the chemical analysis of environmental samples. All designated personnel are to be familiar with this document in order to assure proper interaction between client field operations, laboratory procedures, and data management. Such personnel must attest to an understanding and agreement of this QA management plan by signing the concurrence page at the front of the document.

The QA/QC Coordinator, who is currently also the Laboratory Manager, shall conduct planning, organization, and direction of the Chester LabNet QA program. The overall QA program will be evaluated annually to ensure effectiveness and proper resource allocation.

1.3 Quality Assurance Guidance

This QA Management Plan was written following guidelines provided in the following documents:

- NELAC Quality Systems Revision 16. June, 2003
- NELAC Quality Systems Revision 16. July, 2002
- Quality Assurance Handbook for Air Pollution Measurement Systems, Vols. I and II (U.S. EPA 1976, 1977)
- Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (U.S. EPA 1980)
- NEIC Policies and Procedures (U.S. EPA 1983)
- Contract Laboratory Program Statement of Work for Inorganics Analysis (U.S. EPA 1994)
- Contract Laboratory Program Statement of Work for Analysis of Ambient Air (U.S. EPA1991)
- Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air (U.S. EPA 1994)
- Handbook for Analytical Quality Control in Water and Wastewater Laboratories (U.S. EPA 1979a)
- U.S. EPA Requirements for Quality Management Plans. EPA QA/R-2. (U.S. EPA 1998b)

The Chester LabNet QA Management Plan (QAMP) is intended to be a dynamic document. As new procedures and/or new QA/QC elements become available, the QA Management Plan will be modified accordingly.

2.0 QA Objectives for Data Measurement

2.1 Data Quality Objectives (DQOs)

For environmental laboratory activities, data quality objectives (DQO) may be defined as qualitative and quantitative statements that specify the quality of the data required to support defined analytical requirements (U.S. EPA 1987). Data quality objectives provide the driving force for the level of quality control (QC) required for any analytical task. For example, a field laboratory providing only screening data would have DQOs much less stringent than a laboratory providing data to be used in enforcement actions. Thus the QA Management Plan (QAMP) must be written to provide the level of quality control demanded by the end use of the data.

The paramount analytical requirement for Chester LabNet laboratory is that all measurement data be of the quality required to withstand the scrutiny of litigation. To meet this DQO, the Chester LabNet QAMP is structured to enable the laboratory to provide data of known and acceptable quality. The quality of data is known when all components associated with its derivation are thoroughly documented. Data are of acceptable quality when a rigorous QA/QC program is carried out and the QC indicators fall within predefined limits of acceptability. One of the primary functions of the QAMP is to detail the methods of documentation and to define the mechanisms to be used in attaining data of acceptable quality.

QA/QC requirements vary widely depending on the task being performed and the methodology utilized in performing said task. As such, it is the responsibility of the analysts performing the work to be familiar with the QA/QC requirements of each analytical test performed and to ensure that work they are performing meet these requirements.

2.2 QA Mechanisms for Attaining DQOs

The quality assurance mechanisms used to attain predefined data quality objectives fall with five broad categories: precision, accuracy, comparability, representativeness, and completeness. The characteristics of these mechanisms are defined below.

2.2.1 Precision.

Precision is a measure of mutual agreement among repetitive measurements of the same property. For two measurements (duplicates) the relative percent difference (RPD) will be used to estimate precision:

$$RPD = (difference/mean) \times 100.$$

For more than two measurements, the coefficient of variation (CV, also known as the relative percent standard deviation) will be used to estimate precision:

$$CV = (s/M) \times 100,$$

where s = the standard deviation of the repetitive measurements and M = the arithmetic mean of the repetitive measurements. The precision targets for measurement data are summarized in section 2.3.

2.2.2 Accuracy

Accuracy is the agreement of a measurement (or the average of two or more measurements) with an accepted or "true" value. Accuracy can only be estimated from the results of measurements of samples of known composition. The accuracy estimate will be the percent recovery (%R). For the analysis of standards (initial calibration verifications, continuing calibration verifications, and laboratory control samples), the percent recovery is calculated as follows:

For the analysis of spiked samples, the percent recovery is calculated as follows:

$$%R = [(SSR-SR)/SA] \times 100,$$

where SSR = spiked sample result, SR = sample result, and SA = spike added. The accuracy targets for measurement data are discussed in section 2.3.

2.2.3 Comparability

Comparability is defined as the confidence with which one data set can be compared to another. Comparability in laboratory operations is important in analyzing samples for large projects where sample analysis may occur continuously over many days or may occur sporadically over a long period of time. Comparability will be evaluated primarily on the basis of accuracy and precision estimates. There are no quality control estimators specific to comparability, and comparability must be approached as a data assessment task at a level above that of simply compiling QC statistics. In order to ensure data comparability, Chester LabNet will use standard operating procedures and accepted analytical methods, and data will be reported in generally accepted units of measurements.

2.2.4 Representativeness

Representativeness can be defined both qualitatively and quantitatively and is dependent upon the selection of sampling site and choice of sampling methods. The degree of representativeness is important in planning for the collection of samples and has significant ramifications in the subsequent uses of the data. Sample collection methodology is the most significant contributor to sample representativeness. Unless the laboratory is directly involved in the sampling process, this element of representativeness is beyond the laboratory's control. Chester LabNet will provide assistance to clients to ensure that the sample collection procedures lead to representative data (see section 4.0).

For air sampling, the laboratory can assist in the collection of representative data by minimizing spurious results caused by defective and/or contaminated filter and sorbent media. This is accomplished by acceptance testing filter media and by conducting pre-sampling operations (tare weighing, labeling, packaging, shipping, etc.) in a controlled environment designed to prevent media contamination.

2.2.5 Completeness

Completeness is the amount of valid data actually obtained compared to the amount of data that was expected to be obtained under anticipated sampling/analytical conditions. As in the case for representativeness, the laboratory can assist in sampling completeness by providing air sampling media that have been acceptance tested and have been prepared and shipped to ensure that samples are not lost due to physical deficiencies or contamination.

The analytical component of completeness is controlled by employing qualified, experienced analysts, by adhering to stringent training protocols, and by using written standard operating procedures. The completeness targets for laboratory data are discussed in section 2.3.

2.3 Targets for the DQO Mechanisms

The basis for the targets for the quantifiable DQO mechanisms is that of the U.S. EPA Contract Laboratory Program (U.S. EPA 1990). The default targets are as follows:

Matrix	Sampling Medium	Precision	Accuracy	Completeness
Solid	N/A	<u>+</u> 20%	75 - 125%	99%
Water	N/A	<u>+</u> 20%	75 - 125%	99%
Air	Impinger solution	<u>+</u> 20%	75 - 125%	99%
Air	Filter	<u>+</u> 20%	75 - 125%	99%
Air	Sorbent Tube	<u>+</u> 20%	75 - 125%	99%

Complete directives for all DQOs may be found in the QA/QC section of the Standard Operating Procedure for each analytical technique. DQOs vary widely from one analytical methodology to another; the table shown above is to be considered a general guideline.

DQOs may also vary from project to project, client to client, and from one analytical technique to another. Chester LabNet works closely with the client to ensure that the quality of data generated is of a caliber useable for the client's purposes.

3.0 Chester LabNet (CLN) Organizational Structure

3.1 Organizational Structure and Chain of Command

Chester LabNet is an independent laboratory, owned by 2 private individuals and one large organization (Dusquene Enterprises Inc, Pittsburgh, PA) in a 30-30-40 split, and having no legal ties to any other entity that might have any influence or conflict of interest on the testing performed on site. The laboratory, comprised of LabCor, Inc. DBA (doing business as) Chester LabNet, is legally responsible for work performed at its facilities. Neither management nor the laboratory takes any responsibility for work performed off site by any other entity (such as sampling or final data reporting by clients or other companies). The board of directors is comprised of three people: one representative from Dusquene Enterprises, one of the owners, and the current president of the company. The board of directors has little to no bearing on the day to day operations of the corporation.

Chester LabNet is organized into roughly four departments: Client Services, Gravimetry, Conventional Analysis and XRF analysis. Each of these departments has a series of job titles associated with it. Chester LabNet's management only covers work carried out in the laboratory's permanent facilities. Table 3.0 below outlines each department and its associated job titles and responsibilities.

Table 3.0: Job Titles and Descriptions of Duties.

Department/Title	Organizational Responsibilities
Client Services	Interact with clients in issues regarding business aspects of the laboratory, sample receipt and login, report production/data reporting. Manage internal laboratory systems to meet client's needs.
President	Corporate affairs; liaison with clients for contractual matters; program management; marketing and sales.
Lead Project Manager	Manage day-to-day work flow for projects; coordinate sample receipt and storage, sample analysis, and data reporting activities to ensure project turnaround times; supervise all chain of custody and evidentiary procedures. Primary contact person for client interactions.
Project Manager	Coordinate sample receipt and storage, sample analysis, and data reporting activities to ensure project turnaround times; perform chain of custody and evidentiary procedures; data entry in LIMS; preparation of data reports. Primary contact person for client interactions.
LIMS Administrator	Operation and maintenance (hardware/software) of Laboratory Information Management System (LIMS); creation/editing/validation of report scripts and worklists.
Sample Custodian	Receive samples; fill out chain-of custody forms; interface with client and Laboratory on corrective actions; oversee archiving of analyzed samples.

Gravimetry	Coordinates all filter preparation and weighing to meet client demands as directed by Project Managers
Weighroom	Oversee all weighroom operations and QA/QC, including acceptance
Coordinator/Technical	testing, gravimetry, packaging, shipping and reporting of data; maintain
Director	appropriate inventory levels of filters and supplies.
Weighroom Technician	Perform all weighroom operations and QA/QC, including acceptance testing, gravimetry, packaging, shipping and reporting of data.
Conventional	Perform all chemical analytical work with the exception of XRF analysis.
Analyses	
QA/QC Coordinator	Technical QA/QC oversight; review of protocols, SOPs, logbooks;
	authorize and oversee implementation of all corrective actions; ensure all
	project DQOs and specific QA/QC targets are satisfied.
Inorganics Laboratory	Coordination of all activities within the laboratory; provide technical
Manager	direction to staff and clients; oversee day to day operations of the
	laboratory; supervision and training of all laboratory analysts; oversee
Inorganics Laboratory	maintenance, operation, data compilation, data interpretation, reporting,
Technical Director	QA/QC for all analyses; oversee compliance with all corrective actions.
Lead Chemist	Coordinate day to day operations of the laboratory including flow of work; resource allocations; sample disposal; laboratory hygiene, supplies procurement and instrument maintenance and repair.
Associate Chemist	Analyze samples under the direction of either the Laboratory Manager or Lead Chemist.
XRF	Prepare and analyze samples by XRF.
Senior XRF Scientist/	Oversee instrument operation, maintenance, calibration and repair; data
XRF Technical	compilation, spectral interpretation, QA/QC for all XRF analyses; carry
Director	out corrective actions; supervise and train all XRF technicians.
Associate XRF	Operate and maintain XRFs; XRF data compilation and spectral
Scientist	interpretation; QA/QC for all XRF analyses; carry out corrective actions.

In addition to the brief description above, the QA/QC coordinator shall also:

- serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;
- have functions independent from laboratory operations for which they have quality
 assurance oversight where at all possible (note that due to the size of the laboratory,
 there may be only one employee with both the ability to perform a method, and the
 technical knowledge to oversee that performance. This is a rare occurrence.);
- be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;
- have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system;

- have a general knowledge of the analytical test methods for which data review is performed;
- notify laboratory management of deficiencies in the quality system and monitor corrective
 action. Note that deficiencies are usually corrected by the analyst and/or their technical
 director. Deficiencies are reported to management in such cases where management
 input is required (e.g.capital expenditures) or where a systematic error is discovered.
 This is possible due to the small staffing levels and ease of communication within the
 laboratory.

Due to its small size, most Chester LabNet personnel have two or more titles. All Chester LabNet personnel share responsibility for implementing the Quality Assurance Management Plan (QAMP) and are accountable for those aspects of the program associated with their work areas. The managerial and technical staff is all granted the authority to carry out their duties, identify discrepancies with the QAMP, and initiate corrective actions.

All staff has the authority and responsibility to bring any problems, discrepancies, concerns to the attention of their appropriate supervisor. In situations where privacy is of a concern, all staff has access to all other staff members' home phone numbers. It is understood and encouraged that when needed, employees contact each other at their place of residence. The specific personnel and their titles are listed below:

Table 3.1: Personnel Organization

Personnel	Title
Paul Duda, B.S.	President Lead Project Manager LIMS Administrator
Sheri Heldstab, B.S.	Inorganics Lab Manager Inorganics Technical Director QA/QC Coordinator
Richard Sarver, A.S.	Senior XRF Scientist/ XRF Technical Director
Lisa Ball, B.S.	Project Manager Sample Custodian Weighroom Technical Director XRF Technician
Tony Ochmanek, B.S.	Lead Chemist Weighroom Technician

3.2 Responsibilities and Authorities

The QA organizational structure is provided in Figure 3.1. Extensive cross training allows the uninterrupted flow of samples and data through the entire laboratory process during periods of staff illness or vacations. Vacation days are staggered to ensure no down time will occur. Due to its small size, deputies are not appointed for key managerial personnel. In times of absence, the duties of the manager are performed by the employee with the closest experience level to that manager in the area of knowledge which is needed.

Briefly, the President has oversight over the entire laboratory. He is assisted by the QA/QC Coordinator/Lab Manager, the XRF Technical Director and the Weighroom Technical Director in assuring proper procedures are followed and all data meets predefined QA/QC objectives, including ensuring compliance with NELAC standards where applicable. Any corrective actions will be directed by either the Project Manager, the QA/QC Coordinator, the Weighroom Technical Director, or the XRF Technical Director. All other employees report directly to either the President, the QA/QC Coordinator/Lab Manager, the XRF Technical Director or the Weighroom Technical Director.

Approved signatories for internal laboratory documents may include any member of the staff, but are primarily: the president, the QA/QC director, and any of the four technical directors. Certain documents, such as this one, will require signatures of the full staff. The concurrences page of this document serves as the log of names, initials and signatures for all individuals who may be responsible for signing or initialing laboratory documents or reports.

3.3 Personnel Qualifications and Training

3.3.1 Qualifications

All personnel performing work at Chester LabNet will possess the necessary knowledge, skills, and abilities to perform the work required. No duties or activities will be assigned to staff members not having the qualifications and experience to conduct such work. All personnel performing work at Chester LabNet are degreed professionals. See Appendix E for resumes of all key personnel.

All personnel performing analytical duties possess basic laboratory skills such as the ability to use an analytical balance, to properly read a meniscus line for volumetric work, to use both autopipets and glass fixed volume pipets, to use a burette, to perform basic mathematical calculations including proper canceling of units, to properly identify glassware and its functions, general laboratory vocabulary (such as "buffer", "titrant", "reflux" etc.), fundamental computer

skills (such as saving files, opening software applications, finding files on a computer, etc.) and general laboratory safety.

New personnel are not hired directly by Chester LabNet, but rather through a temporary staffing service. The new employee will remain employed by the temporary staffing company for a period of six months, during which time they are closely monitored for signs of inappropriate or unethical conduct, as well as technical abilities. If any such issue arises, the employee is sent back to the temporary staffing service and a new temporary employee is contracted.

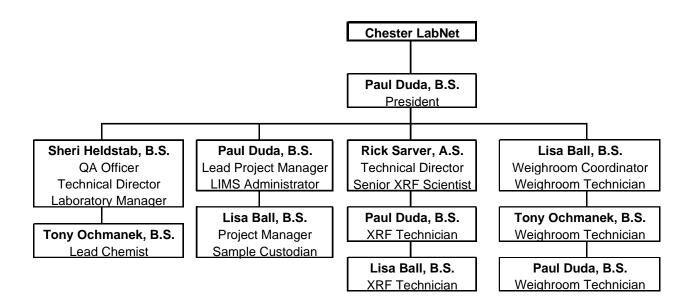


Figure 3.1 Chester LabNet Organizational Structure

3.3.2 Training

3.3.2.1 Technical Training

Chester LabNet has a formal training process delineated in standard operating procedure QA-001, Laboratory Training. This procedure provides for evaluation and documentation of education, previous experience and competency, and three categories of training (on-the-job, briefings, and reading assignments). Training is conducted under the direct supervision of a senior staff person fully qualified in the procedures.

With the exception of method development, no employee will perform any duties without first being trained by a senior employee in that duty. The employee being trained must demonstrate proficiency to the senior employee before being allowed to perform the duty unsupervised. Depending on the nature of the duty, training may take as little as a few minutes, or as much as a month or more. Demonstration of proficiency will vary based on the complexity of the task. For most analytical techniques, the employee being trained will be judged proficient when he or she can replicate the senior employee's data, and/or consistently generate data meeting all the Quality Control parameters of the method.

Training will be considered up to date if the employee has signed off on the most recent version of the test method on an annual basis as documented on the employee's SOP review sheets retained by the Quality Assurance Officer, and has demonstrated at least four sets of QC parameters to be within acceptable control limits (e.g. LCSs). Due to the large number of SOPs, maintaining a training certificate for each and every method a given analyst performs in that analyst's personnel file would be cumbersome at best and ridiculous at worst. In addition, each employee must read and sign off on the latest version of this document on an annual basis.

3.3.2.2 Ethics and Personal Integrity Training

Professional Integrity training takes the form of a three pronged course. The first prong occurs during the hiring process when the employee is still working for the temporary agency. Any signs whatsoever of an employee producing questionable data, or being unduly influenced by any source, be it external or internal, is grounds for immediate dismissal. During the employees first six months as a temporary employee, most issues regarding employee integrity are resolved.

The second prong of professional integrity training occurs during briefing sessions with that employee's supervisor or during the annual integrity training which occurs in conjunction with the scheduled annual safety training. These briefings include the potential ramifications of unethical behavior, to include dry labbing, intentional rolling of clocks, acceptance of bribes or other favors, etc. Chester LabNet prides itself on the quality of data and does not take such behavior lightly. The employee is advised that such behavior will lead to, at a minimum, instantaneous dismissal from the laboratory, and should the issue go as far as legal action by a client, the laboratory will not attempt to

defend the individual, thus leaving them open to possible jail sentences.

The third and final prong of professional integrity training consists of creating an environment where analysts are not punished for variances in data, unexpected results, instrument failure or other unforeseen occurrences which have an effect on the acceptability of the data. Chester LabNet fosters, to the greatest extent it can, an open and trusting environment, where employees may feel free to discuss aberrations openly and without fear of reprisal, such that issues are brought to the fore prior to data being reported, in the hopes that such issues may be appropriately handled and problems reconciled in an ethical manner. Due to the wide variation in air quality samples and requests for odd analyses, the handling of such aberrations will vary greatly, but will always include an agreement with the client as to how the client would like the laboratory to proceed.

All employees of Chester LabNet understand the ramifications of unethical behavior, and by their signatures on this document, attest to knowing the possible outcomes of such behavior. By their signatures on this document, the employees also attest that they are free from any undue pressures or influences which may adversely affect the quality of their work, and will avoid involvement in activities that would diminish confidence in their competency, impartiality, judgment or operational integrity.

4.0 Sampling Procedures

The representativeness of any analytical result can be only as good as the methods used to collect, preserve, store, and ship the sample. Almost always, Chester LabNet has no control over these important steps in the production of analytical data. Whenever possible, the Chester LabNet staff will consult with the regulatory agencies, consultants, or industrial clients to ensure that correct procedures are followed.

The specific sampling procedures recommended by the laboratory are either detailed in the individual protocols and SOPs referenced in this section or are obtained from one or more of the following guideline documents:

- Quality Assurance Handbook for Air Pollution Measurement Systems. Volumes I & II. (U.S. EPA 1976, 1977)
- Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (U.S. EPA 1992a)
- Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from the Atmosphere (U.S. EPA 1992b)
- Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere (U.S. EPA 1992c)
- Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere (U.S. EPA 1997)
- Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods (U.S. EPA 1998c)
- Code of Federal Regulations, various parts, various methods (Source Testing Methods)
- IO Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air (various methods)
- NIOSH Manual of Analytical Methods (NMAM) (various methods)
- U.S. Department of Labor Occupational Safety & Health Administration (various methods)

The sample size, container, preservative, and holding time requirements for the most common analyses performed at Chester LabNet are provided in tables 4.1 (Ambient Air Particulates), 4.2 (Source Emissions), and 4.3 (Indoor Air/Fugitive Emissions). These Tables also contain information on specific methods used (with references), common reporting units, and detection limits.

Sample bottles and containers are usually provided by the client, and the laboratory has little or no control over the appropriateness of the sample containers. On the rare occasion that Chester LabNet provides these containers, they are either purchased precleaned or cleaned in the laboratory following agency-approved protocols (U.S. EPA 1994). Filters are usually shipped and stored in one of the following

containers dependent on the size of the filter: glassine folder inside a manila folder inside a manila envelope, plastic or glass Petri dishes, or plastic Petri slides.

The laboratory has procedures and appropriate facilities for avoiding deterioration, contamination, loss or damage to the sample during storage, handling, preparation and testing. Handling instructions described in appropriate SOPs are followed. When samples have to be stored or conditioned under specified environmental conditions, these conditions shall be maintained, monitored and recorded. Where a sample or a portion of a sample is to be held secure, the laboratory has arrangements for storage and security that protect the condition and integrity of the secured samples or portions concerned.

- Samples are stored according to the conditions specified by preservation protocols, SOPs or published methods, as appropriate: Samples which require thermal preservation shall be stored under refrigeration which is ± 2 °C of the specified preservation temperature unless method specific criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable. (Note that most air quality sampling methods do not require thermal preservation.)
- Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in such a manner to prevent cross contamination.
- Sample fractions, extracts, leachates and other sample preparation products are stored as above, according to the appropriate SOP, or according to specifications in the test method.
- The laboratory has SOPs for the disposal of samples, digestates, leachates and extracts or other sample preparation products.

				DET.	FILTER	FILTER		HOLDING
ANALYSIS	METHOD	REFERENCE	UNITS ^a	LIMIT	SIZE	MEDIUM°	PRESERVATIVE	TIME
TSP	gravimetry	40 CFR 50,	mg	0.1	8X10"	glassfiber or	d	d
		Appendix B				quartz		
PM ₁₀	gravimetry	40 CFR 50,	mg	0.1	8x10"	glassfiber or	d	d
		Appendix J				quartz		
PM ₁₀	gravimetry	IO 2.2	μg	1	37mm	Teflon or	d	d
dichotomous						quartz		
PM _{2.5}	gravimetry	40 CRF 50,	μg	1	47mm	Teflon	none	10 days
		Appendix L					< 4°C	30 days
total	ICP or	40 CFR 50,	µg/cm²	b	various	various	d	d
elements	GFAA	Appendix G						
		IO 3.2						
		IO 3.4						
mercury	CVAA	EPA 7471	µg/cm²	b	various	various	d	d
total	X-Ray	EPA IO-3.3	µg/cm²	b	various	varous	d	d
elements	Fluorescence							
anions	ion chrom.	EPA 300.0	µg/cm ²	b	various	various	-4 °C	d
cations	ion chrom.	EPA 300.4	µg/cm²	b	various	various	d	d
carbon	thermal/	NIOSH 5040	µg/cm ²	0.2	various	quartz	-4 °C	d
species	optical							

 $[^]a$ Units for elements can also be $\mu g/g,\,\mu g/filter,\,\mu g/m^3,$ percent total mass

Table 4.1 Ambient Air Particulates Method Specifications

^b Detection limits are a function of air volume, amount of filter analyzed, and count time (for XRF)

^c Other filter media include cellulose, impregnated cellulose, nylon

^d Not published with method

ANALYSIS	METHOD ^a	REFERENCE	SAMPLE ANALYZED ^b	IMPINGER SOLUTION	PRESERVATIVE	HOLDING TIME
particulates	gravimetry	EPA 5	FH & BH	water	С	С
SO ₂	titrametric	EPA 6	ВН	3% H ₂ O ₂	С	С
NO _x	ion chrom.	EPA 7A	special flask	H ₂ SO ₄ /H ₂ O ₂	С	С
NO _x	ion chrom.	EPA 7D	special flask	H ₂ SO ₄ /H ₂ O ₂	С	С
H ₂ SO ₄ mist	titrametric	EPA 8	FH + impinger	80% isopropanol	С	С
SO ₂	titrametric	EPA 8	ВН	3% H ₂ O ₂	С	С
H₂S	titrametric	EPA 11	ВН	CdSO ₄	С	С
inorganic Pb	ICP or GFAA	EPA 12	FH + BH	0.1N HNO ₃	С	d
part. & gaseous fluorides	ion chrom.	EPA 13B	FH + BH	water	С	С
total reduced sulfur	titrametric	EPA 16A	BH	3% H ₂ O ₂	С	С
HX	ion chrom.	EPA 26	BH	0.1N H ₂ SO ₄	С	С
HX & X ₂	ion chrom.	EPA 26A	ВН	0.1 N H $_2$ SO $_4$ & 0.1 N NaOH	С	С
multi-metals	ICP	EPA 29	FH & BH	HNO ₃ /H ₂ O ₂ & KMnO ₄ /H ₂ SO ₄	С	d
mercury	CVAA	EPA 101	FH + BH	acidic ICI	С	d
mercury	CVAA	EPA 102	FH + BH	acidic ICI	С	d
beryllium	ICP or GFAA	EPA 103	FH filter only		С	d
beryllium	ICP or GFAA	EPA 104	FH & BH	water	С	d
arsenic	ICP or GFAA	EPA 108	FH & BH	water	С	d
ammonia	ion chrom.	CTM-027	BH	0.1N H ₂ SO ₄	4 °C	2 weeks
particulate F & Cl	ion chrom.	CARB 421	FH		С	С
gaseous HF & HCl	ion chrom.	CARB 421	BH	IC Buffer	С	С
total chrome	ICP	CARB 425	FH + BH	0.1N NaOH	С	d
hexavalent chromium	UV/VIS	CARB 425	FH + BH	0.1N NaOH	С	24 hours ^e
multi-metals	ICP	CARB 436	FH & BH	HNO ₃ /H ₂ O ₂ &	С	d
				KMnO ₄ /H ₂ SO ₄	С	d
particulates	gravimetry	Oregon 5	FH & BH	water	С	С
particulates	gravimetry	Oregon 8	FH		С	С

^a Detection limits vary with impinger solution volume, total air volume sampled, etc. Units are mg (gravimetry, titrations, and ion chromatography) or μg (ICP and CVAA).

Table 4.2 Source Emissions Method Specifications

^b FH = front half filter + probe rinse; BH = impinger solutions. For EPA 29 and CARB 436, FH/BH may be combined.

^c Not published with method.

^d By reference (SW-846) mercury is 28 days, and all other elements are 6 months.

^e Post-extraction.

		NIOSH	SAMPLING		HOLDING
ANALYSIS	METHOD	NUMBER	MEDIUM	PRESERVATIVE	TIME
total nuisance dust	gravimetry	0500	37mm TFE or PVC	а	а
respirable particles	gravimetry	0600	37mm TFE or PVC	а	а
arsine	GFAA	6001	hopcalite tube	а	6 days
phosphine	UV/VIS	6002	sorbent tube	а	7 days
SO ₂	ion chrom.	6004	37mm cellulose (front)	а	а
			37mm cellulose (back)		
diborane	ICP	6006	37mm TFE (front)	а	7 days
			sorbent tube (back)		
mercury	CVAA	6009	hopcalite tube a		30 days
Br ₂ & Cl ₂	ion chrom.	6011	25mm Zefluor (front) a		30 days
			25mm Silver (back)		
NO ₂	ion chrom.	6014	sorbent tube	а	7 days
ammonia	ion chrom.	6016	sorbent tube	а	35 days
multi-elements	ICP	7300	37mm TFE or MCE	а	а
chromium (VI)	UV/VIS	7600	37mm PVC a		2 weeks
fluorides	ion chrom.	7902	37mm MCE (front) a		а
			37mm Cellulose (back)		
inorganic acids	ion chrom.	7903	sorbent tube	а	21 days

^a Not published with method.

Table 4.3 Indoor Air/Fugitive Emissions Method Specifications

5.0 Sample Custody and Document Control

5.1 Introduction

When results are expected to be involved in legal proceedings or enforcement, integrity of the sample must be maintained from collection to data reporting. This includes the ability to trace the possession and handling of the samples from the time of collection, through analysis, until final disposition.

Documentation of the sample history is referred to as "chain of custody." This section describes the components of chain of custody and the procedures for their use. Guidance was provided by U.S. EPA (1978a).

A sample is considered to be under a person's custody if it is: 1) in a person's physical possession; 2) in view of the person who has taken possession of the sample; 3) secured by the person so that no one can tamper with the sample; 4) secured by the person so that access is restricted to authorized personnel. Any person who samples or handles samples under chain of custody must comply with the procedures described in this section.

5.2 Chain of Custody Record

5.2.1 External Chains of Custody

To establish the documentation necessary to trace sample possession, a chain of custody record must be filled out at the time of collection and accompany every sample. A chain of custody record is illustrated in Figure 5.1. The record should contain the following minimum information:

- sample Identification (Chester LabNet laboratory identification number or client sample ID)
- sample tag number (if separate tag present)
- site (client sample number or site location identifier)
- signature of sampler
- date and time of sample collection
- type of sample (water, soil, etc.)
- signatures of all persons involved in the chain of custody
- · inclusive dates of possession.
- Analyses requested.

Each person who has custody must sign the chain of custody form. Samples must not be left unattended unless secured and sealed. Note that Chester LabNet has no control over whether or not a client submits a legally defensible chain of custody.

5.2.2 Internal Chain of Custody

Due to its small and secured facilities, Chester LabNet does not utilize internal chains of custody. Samples are at all times kept in a secured part of the facilities, and visitors are not allowed within the confines of the facilities without an escort.

5.3 Sample Labels

Sample labels are necessary to prevent misidentification of samples. Ordinarily, clients will provide their own sample labels, which vary widely in design. An example sample label is illustrated in Figure 5.2. Gummed paper labels or tags are adequate and should include at a minimum the following information:

- client name
- client sample identification number
- site location identifier
- date and time of sample collection
- signature or initials of sample collector
- any preservatives used or matrix of sample if not obvious.

Labels should be affixed to sample containers prior to or at the time of sampling. The labels should be filled out at the time of sample collection. It is recommended that permanent waterproof ink be used when filling out sample labels to avoid damage during shipment, especially where ice or moisture may be present in the shipping container. Note that the laboratory has little to no control over sample labeling as this is performed by the client.

5.4 Sample Seals

Sample seals are used to detect unauthorized tampering of samples following collection and up to the time of analysis, and are usually provided by the client. An example seal is illustrated in Figure 5.2. Seals should be affixed to containers at the time of sample collection, and in such a way as to be visibly damaged should the container be opened. Shipping containers should have custody seals attached to detect possible tampering. Chester LabNet has no control over the correct usage of custody seals.

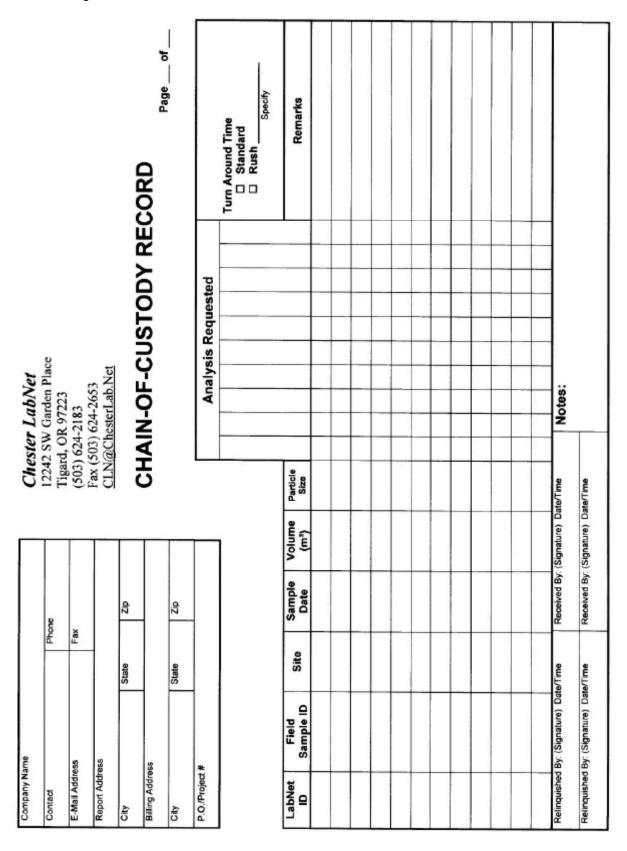


Figure 5.1 Example Chain Of Custody Form

LOT#	
SAMPLE	
ID	
SAMPLED BY	DATE
	TIME
LOCATION	PRESERVATIVE
ANALYSIS	CLIENT

	Custody Seal
Person Collecting Sample	Sample No
	(Signature)
Date Collected	Time Collected

Figure 5.2 Example Sample Label and Custody Seal

5.5 Sample Delivery to the Laboratory

Samples not directly transported to the laboratory by Chester LabNet personnel or by the client must be packaged and shipped according to U.S. Department of Transportation and U.S. EPA regulations, including following DoT HazMat shipping/packaging requirements should they apply. The laboratory has no control and bears no responsibility for the actual packaging and shipping of samples.

Samples should be delivered to the laboratory so that the requested analyses can be performed within the specified holding times. Samples should be accompanied by a chain of custody form (Figure 5.1), however the laboratory has no control over the actions of it's clients. Authorized laboratory personnel will acknowledge receipt of the samples by signing and dating the chain of custody and sample analysis request forms.

5.6 Sample Receiving and Tracking

Sample receiving, log in, and internal identification procedures, including the maintenance of laboratory records, are conducted under written SOPs (see Appendix B). Samples delivered to Chester LabNet are received and inspected at a central location. Shipments are inspected for corresponding samples and sample container integrity. Chain of custody forms are checked against shipment contents. Anomalies are immediately communicated to the client. Sample acceptance criteria and corrective actions if the sample does not meet sample acceptance criteria are given in the appropriate SOPs.

Sample log in is conducted only by the Sample Custodian or a designated alternate. Sample log in is accomplished by entering all sample information into the Chester LabNet Laboratory Information System (LIMS). Upon login, the samples are given a unique laboratory Identification number. The identification is retained throughout the life of the sample in the laboratory. The system is operated so as to ensure that samples cannot be confused physically or when referred to in records or other documents.

The laboratory has a documented system contained in SOP AD-008 for uniquely identifying the samples to be tested, to ensure that there can be no confusion regarding the identity of such samples at any time. Where applicable the laboratory assigns a unique identification (ID) code to each sample container received in the laboratory (in the case of Method 29, more than one container may be received which contains the same sample, or in other cases, more than one bottle of the same sample may be received in the form of "bottle one of two"). This laboratory code shall maintain an unequivocal link with the unique field ID code assigned each sample. The laboratory ID code, in the form of an adhesive label, is placed on the sample container. In cases where the sample collector and analyst are the same individual, or the

laboratory preassigns numbers to sample containers or matrices (e.g. filters), the laboratory ID code may be the same as the field ID code.

The tracking of sample and data flow through the laboratory is a two-tiered process. The LIMS serves as the repository of all physical information about the sample (matrix, sample location, field data, etc.) and the sample identification. The LIMS serves as a progress tracker for the sample by also being the repository of analysis and QA/QC results. Thus the LIMS serves as a database for all sample information and analytical results while being able to provide status, final, and QA/QC reports for clients and in-house staff.

Concurrent with electronic logging, tracking, status, and reporting functions of the LIMS, the Sample Custodian initiates the paper documentation involving chain of custody, QA/QC, and internal tracking via refrigerator logs and laboratory notebooks.

6.0 Chester LabNet Infrastructure

6.1 Facilities and Equipment

6.1.1 Physical Plant

Chester LabNet occupies approximately 3,500 sq. ft. within Building 1 of the Park 217 complex in Tigard, Oregon, a suburb of Portland. The laboratory is of adequate size for the number of analysts and instruments employed and for the protocols in use. The laboratory has all necessary fume, fire, and splash hazard control and response equipment to ensure staff safety, including five fume hoods, a safety shower, and three eye wash stations. Housekeeping is performed as needed by the staff utilizing that particular area of the laboratory, with laboratory management trusting those employees to perform housekeeping in a timely and reasonable manner. Management may address employees who are not keeping their areas clean. Energy sources, lighting and environmental conditions are sufficient to facilitate correct performance of the environmental tests performed on site.

The laboratory includes: an XRF facility; an air filter inspection, weighing, and storage facility with documented controlled temperature and humidity; and a large, general laboratory for inorganics and conventional analyses. The laboratory also includes five fume hoods and approximately 340 linear feet of bench space. Physical separation between neighboring areas in which there are incompatible activities is ensured by the use of dedicated fume hoods and work spaces, dedicated glassware and dedicated pipettes.

Due to the large amount of bench space and small number of personnel, sufficient work space is assured. Access and entryways to the laboratory, as well as aisles and walkways, are kept clear of obstructions at all times for safety reasons. Adequate space is available for sample receipt and storage, chemical and waste storage, and data handling and storage. A laboratory floor plan is provided in Figure 6.1.

6.1.2 Security

Access to the entire building is by cardlock during all hours, seven days a week. During business hours, the main door is kept secured and monitored by a receptionist, who activates an electronic relay to open the door. Visitors are taken into the main laboratory under escort only.

6.1.3 Instrumentation

The laboratory possesses all instruments and accessories needed for the correct performance of the environmental tests, with the exception of extremely outdated, yet still utilized, test methods. Equipment and its software used for analysis are capable of achieving the accuracy required and comply with specifications relevant to the environmental tests performed.

6.1.3.1 Acquisition.

The Chester LabNet current capital equipment inventory is listed in Table 6.1. All equipment used in the laboratory is evaluated prior to use for its ability to ensure consistent high quality. New equipment shall be tested using standards or samples of known concentrations to verify that the equipment is functioning within acceptable parameters. New equipment shall be documented in an equipment specific folder, which shall contain the following information:

- Equipment Operation Verification Study
- Date Received
- Date Installed
- Make, model and serial number of instrument
- Make, model and serial number of any peripherals where applicable.

New equipment shall have a new maintenance log established for that unit. The maintenance logbook will note the date the instrument was brought online into production usage, as well as the other items discussed below.

6.1.3.2 Maintenance.

All instruments have a bound maintenance logbook in which all problems, repairs, and service visits are documented. Every entry in a maintenance logbook shall have the following elements:

- Date and Initials of the analyst making the entry
- Name of person performing the repair or maintenance if different than the analyst
- A complete description of the nature of the problem, symptoms, or preventative maintenance
- Description of parts repaired/replaced/realigned

The description of the maintenance/repair shall be thorough enough that another person reading the entry can identify what the symptoms were (if any), what the suspect parts were (if any) and what steps were taken to repair or maintain the instrument. Also, any hardware or software upgrades will be noted in the maintenance logbook.

Preventative maintenance is scheduled based on guidance from the manufacturer and analyst familiarity with their respective instruments. Preventative maintenance is noted in each instruments maintenance logbook. All primary analysts are responsible for scheduling/performing preventative maintenance on their instruments. Corrective maintenance can be performed either by the primary analyst or by a field service technician, depending on the complexity of the repair needed. All corrective maintenance is noted in the maintenance logbook and the name of the field service technician (if any) is included in the description of the repair.

All support equipment (refrigerators, waterbaths, ovens, balances etc) are maintained in working order. Calibration and/or verification occurs at a minimum annually, depending on the equipment. Maintenance and verification is detailed in appropriate SOPs or within this document. Should a piece of support equipment fall out of specifications, it will be removed from service until such time as it is repaired and/or replaced with a functional unit. Raw data documenting support equipment operation are maintained in various locations throughout the laboratory. Support equipment functioning is verified on an ongoing or as needed basis, depending on the nature and use requirements of the equipment. NIST traceable references are used to verify the functioning of critical support equipment.

The laboratory SOPs detail the use and planned maintenance of measuring equipment to ensure proper functioning and in order to prevent contamination and preventable deterioration of said equipment. Equipment that gives suspect results or has been shown to be defective or outside specified limits will be taken out of service until such time as it has been repaired and demonstrated to be performing properly again. The analyst(s) who operate the equipment are responsible for noting such failures in equipment and initiating the necessary repairs. No data will be released if there is any doubt that the malfunction may cause that data to be non-conforming.

Quality control checks on each instrument are performed during routine analysis. Some of these checks include the metrics of: percent recovery of ICV/CCVs, LCSs and matrix spikes; calibration correlation coefficient; RSDs between duplicate readings of the same

sample; RPDs between duplicate digestions of the same sample. QC metrics vary from instrument to instrument and from method to method. Non-measured quality control checks are performed by the analyst as a routine aspect of running the instrument. These checks may include, but not be limited to: visual inspection of sample introduction systems (e.g., graphite tube wear, nebulizer blockage, spray chamber cleanliness); auditory observations of instrument functioning (e.g., unusual noises etc), visual monitoring of autosampler etc.

6.1.3.3 Calibration

For all laboratory instrumentation (both support and instrumental), the calibration procedures used are those given in the manufacturer's instruction manuals, in the relevant agency protocol guidance documents (e.g., U.S. EPA 1994), or more specifically in the SOPs. All instruments are demonstrated to be capable of attaining and maintaining calibration during protocol validation. Instruments are then recalibrated at regular intervals as specified in Standard Operating Procedures (SOPs).

For inorganic and conventional analyses, instruments will be calibrated using a blank and a series of standards as outlined in the respective SOPs. Working calibration standards are prepared by diluting high-concentration prepared or commercial stock solutions. Wherever possible, commercial standards will be used that are traceable to National Institute of Standards and Technology (NIST) standard reference materials. All analytical instruments within the inorganics laboratory are calibrated immediately prior to use (ICP, GFAA, CVAA), with the exception of the IC, which is calibrated on an as needed basis when continuing calibration verification standards fall outside of control limits.

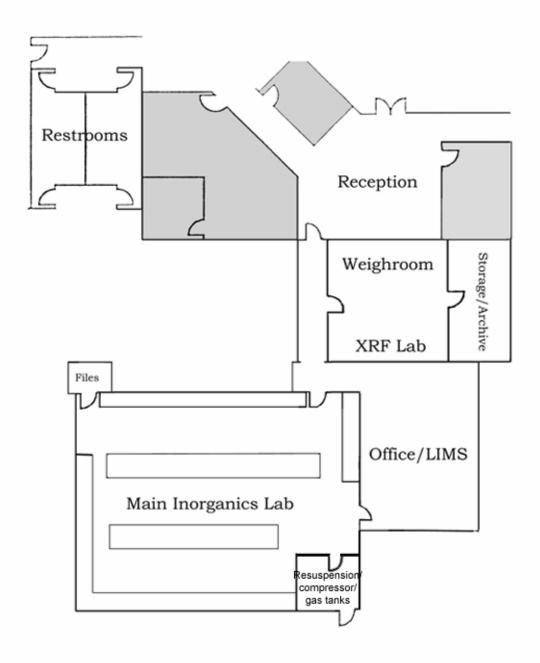


Figure 6.1 Chester LabNet Floor plan

X-Ray Fluorescence Spectrometer Kevex Model 770 with advanced computer interface

by IXRF, Inc. (1997)

X-Ray Fluorescence Spectrometer Kevex Model 770 with advanced computer interface

by IXRF, Inc. (2001)

X-Ray Fluorescence Spectrometer Kevex Model 770 with 771 upgrade (2001)

Organic/Elemental Carbon Analyzer Sunset Laboratories Model (2001)

Plasma Emission Spectrometer PerkinElmer Optima 2000 with autosampler (2002)

Graphite Furnace Atomic Absorption Spectrometer Perkin-Elmer Aanalyst 600 with autosampler (2002)

lon Chromatograph (2) Dionex Model DX500 with autosampler (2000, 2004)

Mercury Cold Vapor Spectrophotometer (2) Bacharach Model MAS-50D with advanced computer

interface (1999, 2001)

UV/VIS Spectrophotometer Milton Roy Spectronic 20D (1989)

Turbidimeter Hach Model 22100A (1989)

Conductivity Meter Curin Matheson LabCraft (1989)

pH/mV Meter Orion Model 720A (1992)

General Laboratory Analytical Balance AND Model ER-182A (1989)

Gravimetry Laboratory Analytical Balance Sartorious Model B120S with dedicated computer (1988)

Gravimetry Laboratory Microbalances (2) Two Cahn Model C-31's with dedicated computer (1989)

Laminar Flow Hoods (2)

Atmos-Tech and LabConCo, both with prefilter and

HEPA finish filter

Laboratory Oven American Scientific Products Model DN-43 (1990)

Muffle Furnace American Scientific Products Model FP-41 (1990)

Laboratory Information Management System (LIMS)

Northwest Analytical, Version 1.65K resident on a

PC computer operating Windows NT (1998)

Table 6.1 Chester LabNet Capital Equipment Inventory

(Acquisition date in parentheses)

For XRF analysis, fundamental calibration is via commercial, single-element, thin-film, vapor-deposited standards (from MicroMatter, Inc.) and via organo-metallic acetate film standards containing two or more non-interfering elements (from U.S. EPA). This calibration is performed on an as needed basis when continuing calibration verification standards fall outside of control limits. Calibration verification is via NIST thin film standards SRM 1832 and SRM 1833 for eleven representative elements.

Calibration is verified whenever possible by the analysis of independent standards. Whenever possible, U.S. EPA control limits for percent recovery of calibration verification standards will be used (e.g., 90-110% for metals by ICP). Criteria for the acceptance of an initial instrument calibration are indicated in the appropriate SOPs. In addition, continuing calibration standards will be analyzed at regular intervals throughout any analysis run to demonstrate the absence of significant instrument drift. Again, whenever possible, U.S. EPA control limits for percent recovery of continuing calibration standards will be used (e.g., 90-110% for metals by ICP).

For inorganics and conventional analyses the results of the analysis of initial calibration verification and of continuing calibration standards are tabulated and included in the data report to the client.

The weighroom Sartorius analytical balance is calibrated once daily with a certified weight. The weight is certified annually by an outside service and the certification receipt is maintained in a 3 ring binder in the weighroom. Calibration is checked with a different weight. The check weight is not certified as it is used primarily as a check of precision to verify that the calibration weight, and thereby calibration, has not changed. Throughout analysis, a zero reading is taken every 10 weighings to verify calibration.

The weighroom Cahn micro-analytical balances are calibrated prior to weighing each set of 10 filters. The weight is certified bi-annually by an outside service and the certification receipt is maintained in a 3 ring binder in the weighroom. Calibration is checked with a different weight. The check weight is not certified as it is used primarily as a check of precision to verify that the calibration weight, and thereby calibration, has not changed. Throughout analysis, a zero reading and a mass reading is taken every 10 weighings to verify calibration. The balance is also fully recalibrated every 10 weighings during a sequence of weighings.

The laboratory balance is calibrated once daily as needed with a certified weight internal to the balance. The balance/weight is certified annually by an outside service and the certification receipt is maintained in a 3 ring binder in the weighroom. Calibration is checked with a variety of weights, most commonly a 5mg, 50mg, 500mg and 100g weight. These weights are not certified annually as they are used primarily as a check of precision to verify that the internal calibration weight, and thereby calibration, has not changed.

Both the weighroom and laboratory balances, as well as any certified weights used during the calibration, are clearly marked as to the status of the calibration, including the date when the calibration has expired, such that recalibration will be performed within the calibration due date.

Pipette calibration is verified monthly and pipettes are adjusted as needed to fall within 1% of their set value. For further detail, refer to SOP QA-007.

Where possible, sufficient raw data records are retained to permit reconstruction of the initial instrument calibration, to include calibration date, instrument, analysis date, analyte name, analyst's initials, concentration and response, calibration curve or response factors, and/or the mathematical means by which raw data is reduced to final data. All sample results are calculated from the initial instrument calibration unless otherwise indicated in the SOP or method. Where calibrations give rise to a set of correction factors (e.g. XRF), the laboratory has procedures to ensure that copies (e. g. in computer software) are correctly updated contained within the appropriate SOPs.

6.1.3.4 Equipment Records

Records are maintained for each major item of equipment and its software used for testing. The records include at least the following information, where known:

- the identity of the item of equipment and its software;
- the manufacturer's name, type identification, and serial number or other unique identification;
- checks that equipment complies with the specifications;
- the current location, where appropriate;
- reference to the location of manufacturer's instructions, if available;

- dates, results and copies of reports and certificates of all calibrations, adjustments, acceptance criteria, and the due date of next calibration where applicable;
- the maintenance plan, where appropriate, and maintenance carried out to date; documentation on all routine and non-routine maintenance activities; and reference material verifications.
- any damage, malfunction, modification or repair to the equipment.
- date received and date placed in service (if available);
- if available, condition when received (e.g. new, used, reconditioned);

6.2 Supplies and Service Procurement

6.2.1 Sampling Media

Filters are purchased in bulk and stored in the temperature- and humidity-controlled weighroom. Before use, filters are visually acceptance tested using the following rejection criteria:

- Pinholes
- Creases
- Tears
- Thin or thick spots.

For smaller filters with support rings, the following additional rejection criteria apply:

- Detachment from mounting rings
- Warped mounting rings.

The filter surface is inspected for loose particles, which, if found, are blown off using a simple aspirator bulb. If there is a significant rejection rate for any given filter lot, the entire lot is rejected and returned to the manufacturer.

In addition to the physical acceptance criteria described above, the following combination of manufacturer acceptance criteria and laboratory QC criteria must be met for all 8x10" quartz filters:

Acceptance or QC Criterion	<u>Value</u>
Collection efficiency	99.998% for a 0.3mm particle @ 5 cm/sec face velocity
Alkalinity	< 20 µeq/g

Integrity acceptance	<u>+</u> 1.9 μg/m³
Equilibration temperature	21 <u>+</u> 3 °C
Equilibration humidity	35 ± 5% relative humidity

where filter integrity is defined as the maximum allowable variation in weight for a blank quartz 8x10" filter after 24-hour equilibration at controlled temperature and humidity. This test must be met for filters shipped to the field and is based upon a maximum allowable variation at the balance of ± 3 mg per unexposed filter and assumes a nominal air sampling volume of 1,600 m³.

Chester LabNet keeps some sorbent tubes in stock, and occasionally purchases other non-filter sampling media. These media are shipped to the client as received from the vendor, with no acceptance testing being performed due to the consumption of the material during testing. If acceptance testing is required under contractual obligation, the laboratory will acceptance test one unit of media per lot number (or other as required by contract). Otherwise, Chester LabNet makes no claims as to the quality of the material. Provision of non-filter sampling media is performed merely as a service to the client, and on an as-requested basis only.

6.2.2 Standards

Commercially prepared, NIST-traceable primary standards are used for all non-XRF elemental analyses and all ion analysis. The certificate of analysis, where supplied, for each standard is stored in a 3-ring binder stored in the main laboratory.

Single- or multi-element standards are used to prepare working calibration solutions. Multi-element standards are used to prepare working calibration verification solutions. Whenever a working standard solution is prepared, all pertinent information is entered into the appropriate bound standards logbook stored in the main laboratory. Information in the logbook includes: reference to the primary standard lot number for the primary standard used; concentration of the primary standard; concentration of the working solution, matrix of the working standard, and any dilutions of this working solution.

Standard weights used to calibrate the balances are certified annually to be within ASTM class 1 tolerances, and are used solely for the purpose of calibrating the balances. Standard weights used to verify calibration of balances are not certified as they are used solely as a test of precision. Control limits are set based on previous measurements of the verification weights.

6.2.3 Digestion Reagents

All digestion reagents (acids and hydrogen peroxide) are purchased from vendors with known reliability. Acids and peroxide are, at a minimum, reagent grade. Currently Trace Metals grade acid and peroxides are being used for digestion of samples requiring non-XRF metals analysis. The manufacturer certifies that trace levels of analytes are below any level of concern for the intended use by the laboratory. Reagent procurement is described in greater detail in SOP AD-005.

6.2.4 Reagent (DI) Water

Reagent water is manufactured onsite using a U.S. Filter pretreatment system, followed by a Barnstead Nanopure polishing system. The manufacture of Reagent or Deionized (DI) water is discussed thoroughly in SOP AD-006. Briefly, water is generated using the system noted above. At the time of production, the resistivity of the water is measured by the Deionizing system, and the resultant measurement is recorded in a DI Water Control Chart kept near the system. Water is generated in small quantities and is stored in quantities not to exceed 15 gallons. The conductivity of the stored water is not measured daily due to the high turnover of stored water.

Some methods specify the ASTM Type rating of water to be used during analysis. The ASTM specifications for reagent water include total matter, electrical conductivity, electrical resistivity, minimum color retention, soluble silica, method of preparation and intended use. For the intended uses at Chester LabNet, both Type I and Type II are applicable:

- ASTM Type I: "...shall be used where maximum accuracy and precision is indicated, provided dissolved organic matter is not a possible interference."
- ASTM Type II: "...shall be used for most analytical procedures and all procedures requiring water low in organics."

The difference in method of preparation between ASTM Type I and Type II are as follows:

- ASTM Type I: distillation of feed water followed by mixed bed ion exchange polishing followed by a 0.2 μm finishing filter (<u>feed water</u> must have a conductivity ≤ 20 μmhos/cm at 25 °C)
- ASTM Type II: distillation of feed water such that the <u>distillate</u> has a conductivity < 1.0 µmho/cm at 25 °C, recognizing that to meet this criterion the feed water may have to be treated by distillation (essentially making it a two stage distillation process), ion-

exchange, or reverse osmosis (there is NO feed water criterion and NO requirement for a finishing filter).

Of the measure specifications, only resistivity is monitored at Chester LabNet:

ASTM Type I: ≥ 16.67 megohm/cm at 25 °C

ASTM Type II: ≥ 1.0 megohm/cm at 25 °C

Technically speaking, the DI water prepared at Chester LabNet is not strictly either ASTM Type I or Type II, but rather a hybrid missing the specification of distillation. Typically, the resistivity of DI Water at Chester LabNet is greater than 17.5 megohm at the time of production. Due to gaseous CO₂ being absorbed into the water from laboratory air, resistivity measurements after the time of production may be less than 16.7 megohm, however, no analyses are affected by this dissolution of CO₂.

6.2.5 Service Procurement

A plan for routine inspection and preventative maintenance is followed. Scheduling of specific preventative maintenance programs is based upon identifying critical components and maintaining an appropriate spare parts inventory. Each instrument has a maintenance logbook in which all problems, repairs, and service visits are documented. The maintenance logbooks are stored in the main laboratory next to the appropriate instrument.

Most frequently used services are provided by companies with known reputations and are procured from the manufacturers when possible. For servicing of instruments where the analyst cannot repair the failure on site, the instrument manufacturer's field service technicians are called. For the ICP and GFAA servicing, this is primarily the Perkin-Elmer. IC servicing is performed by Dionex field technicians. OC/EC servicing is performed by Sunset Laboratories. The CVAA is shipped back to Bacharach for servicing and repair, and the backup CVAA is used while the other instrument is being serviced. XRFs are serviced by Thermo Noran. Balances are serviced annually by Quality Control Services, which is also the company responsible for recertifying all weights used in the calibration and verification of the balances. The weighroom HVAC system is serviced and repaired by Portland Mechanical Contractors, who were responsible for the installation of the system when the facilities were first brought online.

6.2.6 Evaluation of suppliers

Due to its size and the ease of communication within and between departments, Chester LabNet does not have a formal evaluation process for suppliers of goods or services which may affect the quality of environmental testing performed at the laboratory. A list of suppliers is maintained by the purchasing agent (president). The suppliers utilized by Chester LabNet have a long standing record of providing goods and services which meet or exceed the laboratory's needs.

Should a change be noticed in the quality of reagents or services, or a change in instrumentation require a change in reagents, a new vendor and/or brand is utilized. These reagents are quickly checked to ensure they meet the needs of the laboratory, however, due to the large volume of reagents used, this check is not documented. A change in the supplier and/or manufacturer of critical consumables is extremely rare.

6.3 Computer Hardware and Software

6.3.1 Computer Hardware

All instruments except the weighroom balances and the cold vapor mercury analyzer were purchased with computer hardware supplied by the instrument manufacturers. The weighroom balances and cold vapor mercury analyzer are controlled with personal computers equipped with general I/O interface boards, purchased on the open market.

Computers used for report generation, data manipulation and other office needs were purchased on the open market. All office computers, and several of the instrumental computers are networked using a LAN hub. These computers also have internet access through the LAN.

All computers are maintained to assure proper operation and are provided with the environmental and operating conditions necessary to keep the computer in functional condition.

6.3.2 Computer Software

The ICP and GFAA use Perkin Elmer proprietary software for instrument operation and generation of analysis results. The CVAA uses Bacharach proprietary software (MercuReport II) for data collection and generation of analysis results. The IC uses Dionex proprietary software for instrument operation and generation of analysis results. Two of the XRFs use IXRF proprietary software for data collection, the third XRF uses Kevex proprietary software for data collection. All XRF data manipulation is performed using Chester LabNet proprietary MS Excel workbooks. Filter gravimetry software was programmed by an independent contractor specifically for Chester LabNet. The LIMS software package was purchased from Northwest Analytical of Portland, Oregon. All other software used is of the 'off the shelf' variety.

Validation of all software except the proprietary gravimetry software is considered by the laboratory to have been validated by the manufacturers of the software. The filter gravimetry software was validated by simple visual comparison with the balance face readouts and by hand checks of the calculations it performs prior to being brought online in May 2002. This software was documented by the outside consultant who wrote it.

6.3.3 Backup of LIMS System

The LIMS system is backed up to a separate hard drive daily after the close of each business day. Once per week, the entire system including the LIMS and company financials are fully backed up to CD-RW and the backup CD is kept off site. See SOP AD-007 for further details.

7.0 Analytical Procedures

7.1 Analytical Method Selection

Analytical method selection is performed by clients of Chester LabNet (CLN). CLN adheres to the methods chosen for each analyte by following written approved Standard Operating Procedures (SOPs) in all analytical endeavors. Where possible, Standard Operating Procedures (SOPs) are based upon other agency's approved methodologies, such as the U.S. EPA or NIOSH. See Appendix C for a listing of most commonly run methods.

Method references may include the following:

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (U.S. EPA 1996)
- Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air (U.S. EPA 1998a)
- NIOSH Manual of Analytical Methods (NIOSH 1994)
- Source Sampling Manual, Volume I (Oregon DEQ 1992)
- Code of Federal Regulations, part 50 (U.S. Federal Government)

The laboratory will follow the published method to the extent possible. In many cases, the method may be so outdated as to make following it to the letter impossible (e.g. requirements to use instrumentation no longer available). In all cases, relevant QC will be at least a stringent as that required by the method. Regular deviations from the published methods shall be described in the relevant SOP, along with the technical justification for any departures. Some clients are already familiar with the need for departures from the method, other clients are educated as to the need and form these departures take. The laboratory will inform the client if a better method is available to achieve their desired result, or if the method the client is requesting may not be suitable for the client's purposes, however the client has the ultimate decision making in which method is to be used for analysis.

7.2 Standard Operating Procedures

All laboratory and instrumental procedures used by Chester LabNet follow written Standard Operating Procedures (SOPs, see Appendix B) following the guidance in EPA (1995). These SOPs are either written in-house by the employee most familiar with the method or by a person designated as the technical writer working closely with that employee, and are taken through a multistage review process prior to being approved for general laboratory use.

SOPs are stored in three ring binders in the clerical area of the laboratory. They are organized into three ring binders based on their applicability to various tasks performed within the laboratory. New SOPs are given a number in chronological sequence to those already existing within that department (see appendix B).

The SOPs written by and unique to LabNet cover facility and laboratory operations such as gravimetric analysis, sample log in, building security, etc. and are provided upon request. The SOPs used exactly as provided by recognized authorities are included in the QA Plan by reference in Tables 4.1 through 4.3. Occasionally, a published protocol will have been changed at Chester LabNet to improve overall method precision, accuracy, or detection limits or to take advantage of a new technology.

For these protocols and for many in-house protocols, the written SOPs will contain the following elements:

- Standard Operating Procedure Cover Page (number of last revision and signatures of review)
- Review History Page (including a chronology of revisions and date of last revision)
- Section 1.0: Introduction
 - o Reference Method
 - o Applicable matrices
 - o Detection limits
 - o Method Performance
- Section 2.0: Summary
 - Scope and Application
 - o Summary of Method
 - o Interferences
 - Sample collection/preservation/shipment/storage
- Section 3.0: Safety
- Section 4.0: Pollution Prevention and Waste Management
- Section 5.0: Apparati, Equipment and Supplies
- Section 6.0: Reagents and Standards
- Section 7.0: Preparation, Calibration and Standardization
- Section 8.0: Procedure
- Section 9.0: QA/QC
 - Frequency
 - QC statistic
 - Control limits (including exceptions)
 - Corrective actions
 - Notes (optional)
- Section 10.0: Calculations
- Section 11.0: References
- Section 12.0: Definitions
- Section 13.0: Analyst's Notes
- Figures and Tables as appropriate

After an analytical procedure is developed and becomes a laboratory method, a formal standard operating procedure (SOP) is written to thoroughly describe the methodology. The document will be

written by the employee who developed the procedure, the QA/QC Coordinator or a technical writer designated by the laboratory manager who will compose the document under the guidance of the originating analyst. Upon completion of a draft procedure, the author of the SOP signs and dates the cover page and turns the SOP over to a technical reviewer. The SOP is then reviewed, in turn, by the technical reviewer, and the QA/QC Coordinator. The purpose of each review step is to ensure completeness, technical accuracy, clear and concise writing, and adherence to formatting. Frequently, one individual may sign the document more than once if it is appropriate to do so. For example, the Author and QA/QC Coordinator may be the same person, as is the case with this document. A minimum of two signatures is required for all SOPs. Once all review personnel have signed off on an SOP, an effective date is clearly marked at the bottom of the title page.

The QA/QC coordinator will review SOPs on an annual basis and in consultation the appropriate technical director. When it is determined that enough modifications and/or additions warrant, a new version of the SOP will be written. New editions are not issued for simple corrections such as typographical errors or formatting issues. The annual review page will be signed and dated by the person performing the review. If another version of an SOP is written. The newly retired version is now stapled along the right margin and retained in the archived SOPs file drawer. The newest version is placed loose in the three ring binder.

Any SOP which has not been used within the past 5 years is labeled "suspended" by the QA Officer during the annual review, as long as the laboratory still has the ability to perform the method. Should that method be requested by a client, a review of the old SOP will occur before analytical work begins. Any SOP for which the laboratory is no longer capable of performing the work (e.g. broken instrumentation was never replaced, or the method is cost prohibitive for the number of samples, etc.) is labeled "deactivated" by the QA Officer during the annual review. Deactivated and Suspended methods are not reviewed during the normal annual review cycle.

SOPs are used for training and as a reference and audit tool. The signed original SOP is placed in one of several three ring binders in the office area of the facility. Copies are made only for proposals and per the QA/QC requirements of specific projects. All copies are stamped "COPY" in red across the signature portion of the cover sheet. No other copies are made or distributed throughout the laboratory. For greater detail, refer to CLN SOP QA-003 (see Appendix B)

7.3 Review of Laboratory Capabilities for Requests', Tenders' and Contracts' Methodologies

For the majority of their clients' needs, Chester LabNet uses the following processes for determination of whether or not work requested by clients is feasible. Given the proliferation of agencies covering Air Quality Analysis (CFR, EPA, NIOSH, OSHA, CARB etc) and the multiplicity of challenges faced by field samplers and Air Quality Engineers specific to each source sampled, the laboratory may or may not be easily capable of performing a new analysis. For formal contract work, the review process, including any variances in the methodology, is documented, usually in the form of a memorandum or email, and that documentation is kept with other contract documentation.

If the method requested is a method which the laboratory runs with some degree of regularity, the project manager may simply tell the client that we do have the capabilities of performing the method based on prior experience with that analytical technique.

If the method is an entirely new method to the laboratory, the project manager will request a copy of the method from the governing agency from whom it was issued. The project manager will then give the copy of the method to the technical director of the department which would be involved in the analysis of the samples. The technical director will read the method thoroughly to assure that the laboratory has the technical knowledge, instrumentation, equipment and capability to analyze the samples in a manner compliant with the method.

If the method is a completely new method (e.g. not a mirror of another method found elsewhere, as is the case with many Air Quality methods where, for instance, a CARB method is nearly identical to a CFR method), the technical director is responsible for ensuring, usually via a test run with known standards, that the laboratory is capable of performing the method. Some newer methods may be simple enough that a test run is not necessary. These methods are generally gravimetric methods, with great similarities to other forms of gravimetric work. Minor variations of the method, if needed, would be discussed with the client and agreed upon in advance of any samples being sent to the laboratory. After the method review and, if needed, a test run of the method, the technical director will then notify either the project manager or the client directly of the laboratory's ability to perform the method.

In the cases of non-conforming methods, the technical director of the department involved will be asked to speak directly to the client to determine and come to agreement on exactly how the samples are to be analyzed. In this scenario, the technical director will then be able to inform the client of the laboratory's ability or inability to analyze the samples in a manner compliant with the client's wishes. The client's desired methodology will be documented within the data report.

Chester LabNet QA Management Plan

In all cases, requirements of the contract must be adequately defined, documented, and understood, by both the client and the laboratory. Any and all differences between the request and contract are resolved prior to any work starting. If a contract is amended after work has already been started on the project, the same review process is utilized.

All documentation pertaining to a specific contract or accreditation, including contracts, CARs, correspondences etc, are retained in client/project specific folders by the laboratory president.

7.4 Laboratory Developed Methods

All methods developed in house for whatever reason will have an associated SOP. The in-house methods will undergo the same annual review cycle as all other SOPs. Methods developed in house span the range of dishwashing to filter impregnation. In some cases, methods may be developed to fill in gaps found in other published or promulgated methods (e.g. inspection of filter media, impregnation of media etc).

7.5 Non-standard Methods

Non-standard methods are rarely employed by Chester LabNet, however, when an occasion arises in which the employment of a non-standard method is needed, the method will be developed in conjunction with the client. These methods are almost never used by any other client, or for any other project, and tend to be a one-time contingency need. As such, full validation of the method may not be possible, and presents an undue burden on the laboratory. Such analysis will be documented fully, all directives issued by the client will be noted in the data file, and attempts are always made to get written confirmation from the client as to the acceptance of the proposed methodology. Documentation by the analyst will include specifics, where not obvious, pertaining to the analysis.

The acceptance/rejection criteria for non-standard methods default to methods of similar chemistries or to CLP guidelines if at all possible. When not possible, acceptance/rejection criteria may be based upon Precision and Bias studies and/or IDL/MDL studies.

7.6 Validation of Methods

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Chester LabNet considers methods published or promulgated by established authorities (e.g. EPA, NIOSH, OSHA, state environmental agencies etc) to be previously validated by the issuing authority.

Non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope and amplifications/modifications of standard methods are rarely performed at Chester LabNet. In such cases as these methods are performed, the client is notified as to the status of the validation of the method. It is then the client's option to use a non-validated method, or to require validation of the method. In cases where there client requests complex validation of a method, the laboratory will frequently charge a method development fee. The client will be notified if the range and accuracy of the developed method is not relevant to the client's use, however, the client shall have the final decision as to whether to proceed or not. Any client wishing to proceed with a method deemed by the laboratory to be insufficient to meet the client's needs shall receive notification of that opinion in the case narrative within the data package.

7.7 Estimation of Uncertainty of Measurements

Chester LabNet does not perform any work involving the issuing of calibration certificates, therefore, under NELAP criteria, the laboratory is not required to have procedures in place for estimation of uncertainties for methods employed by the laboratory. Only two methods utilized at the laboratory issue uncertainties as part of standard reporting formats: analysis of metals by XRF, and analysis of Carbon by OC/EC. A full explanation of determination of uncertainty for measurements by XRF is contained within SOP XR-005. Determination of uncertainties for OC/EC is performed by the instrument software.

Should a client request uncertainties for a particular method, efforts will be made to identify the most significant contributions to the uncertainty of the method, and to determine the uncertainties based upon historical results for that particular method and analyte. Note that due to the extremely wide variation in sampling, media and analytes requested by clients, it is quite possible for upwards of 4 years to elapse between analytical events utilizing the same media and processes. In essence, unless the method is very commonly performed, it is possible that the laboratory will not be able to estimate uncertainty with real world data.

Where possible, uncertainties would be calculated be determining the three sigma value of at least 20 LCS standards run over a period of time not to exceed one year. If 20 LCS standards have not been run for a particular method within a one year time-span, all LCS values obtained within the previous 12 months shall be used. These standards would be analyzed using the same preparatory and analytical techniques as utilized for sample analysis.

Normally, the greatest factor contributing to uncertainty is the field sampling portion of the method, which the laboratory has no control over. Other significant sources of uncertainty in methods employed by Chester LabNet are: the preparation of samples, sample matrices (filter contamination or incorrect matrix), sample composition (uneven sample deposits on filters), and instrumental uncertainty. In all cases, the uncertainty would still be calculated based upon results of a known standard.

8.0 Quality Control and Corrective Action Procedures

8.1 Introduction

Quality Control (QC) consists of the operations used to ensure and document instrument performance and the precision and accuracy of a result. These operations vary from instrument preventative maintenance to the analysis of independent standards. Corrective action procedures are designed to maintain instrument performance and precision and accuracy within predefined limits and are initiated whenever these limits are exceeded. Typical corrective actions are recalibration, reanalysis, redigestion, etc. The specific QC checks used at Chester LabNet and their control limits are patterned wherever possible after those used in the U.S. EPA Contract Laboratory Program (U.S. EPA 1986, 1994) and are detailed in the specific analytical SOP.

The laboratory has quality control procedures for monitoring the validity of environmental tests. This monitoring is planned and reviewed and may include, but not be limited to, the following:

- regular use of certified reference materials and/or internal quality control using secondary reference materials;
- participation in interlaboratory comparison or proficiency-testing program (primarily used in XRF analysis and major contractual work)
- replicate tests or calibrations using the same or different methods;
- retesting or recalibration of retained samples;
- correlation of results for different characteristics of a sample (for example, total chromium should be greater than or equal to hexavalent chromium).

Where possible, Chester LabNet ensures than the applicable QC standards are addressed as follows:

- Chester LabNet has detailed written protocols in place to monitor the following quality controls:
 - o positive and negative controls to monitor tests such as blanks, spikes, LCSs, etc;
 - tests to define the variability and/or repeatability of the laboratory results such as duplicates or replicates;
 - measures to assure the accuracy of the test method including calibration verifications and/or continuing calibration verifications, use of certified reference materials, proficiency test samples, or other measures;
 - measures to evaluate test method capability, such as detection limits and quantitation limits;
 - selection of appropriate formulae to reduce raw data to final results;

- o selection and use of reagents and standards of appropriate quality; and
- measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the test method such as temperature, humidity, light, or specific instrument conditions;
- All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance criteria are used to determine the usability of the data.
- The laboratory has procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist.
- The quality control protocols specified by the laboratory's SOPs are followed. The laboratory
 ensures that the essential standards outlined in mandated methods or regulations (whichever are
 more stringent) are incorporated into the SOPs. When it is not apparent which is more stringent
 the QC in the mandated method or regulations is to be followed.

8.2 Quality Control Operations

Quality control operations are broadly classified into those designed to monitor instrument performance only and those designed to monitor the entire sample collection and analysis process. QC operations designed to monitor the representativeness of sampling methods are beyond the scope of this document. The extent to which the factors contribute to the total uncertainty of measurement differs considerably between various environmental tests. Each SOP indicates the QC parameters applicable to that particular procedure. Below is a brief summary of the more common QC parameters. Note that not all methods are capable of having these checks performed (e.g. pH measurements will have no blanks). The following QC parameters are followed as closely as possible based on the methodology being used. Refer to individual SOPs for further detail of required QC elements.

8.2.1 Instrument Performance QC

Instrument performance is initially optimized by following written calibration and preventative maintenance procedures and is monitored during instrument use by analyzing QC check samples. See Section 7.0 of this QA Plan for a description of the first two QC elements. The frequency of analysis of instrumental QC checks is related to the number of "analytical" samples run in a single, continuous sitting at the instrument, and is covered in depth in the respective instrumental SOPs. The preparation of QC check samples, the pertinent QC statistics, the calculation methods for these statistics, and the corrective actions for out of control QC checks are also contained in the SOPs. The specific checks are as follows:

- Initial Calibration Verification Standard (ICV) analyzed immediately after initial
 calibration; percent recovery limits are as published in U.S. EPA (1994) or as specified by
 the client; for results outside of control limits, terminate analysis, correct problem,
 recalibrate if necessary, and reanalyze ICV.
- Initial Calibration Blank (ICB) analyzed immediately after the ICV; where the absolute value of the result is above detection limits, terminate analysis and recalibrate the instrument.
- Continuing Calibration Verification Standard (CCV) analyzed immediately prior to the first CCB and again prior to the last CCB at the end of the analytical run; for greater than ten samples, must also be analyzed at a frequency of 10 percent immediately prior to the CCB; percent recovery limits are as published in U.S. EPA (1994) or as specified by the client; for results outside of control limits, terminate analysis, recalibrate if necessary, and reanalyze any samples affected by the recalibration. In instances where the instrument is not calibrated immediately prior to analysis, a CCV will be analyzed prior to the analysis of any samples to demonstrate that calibration is still within control.

The following elements of continuing calibration verification analysis are essential, with minor variances for some analyses being noted in the appropriate SOPs:

- The details of the continuing instrument calibration verification procedure,
 calculations and associated statistics are included in the test method SOP.
- A continuing instrument calibration verification will be analyzed at the beginning and end of each analytical batch. The concentrations of the calibration verification fall within the established calibration range.
- Sufficient raw data records are retained to permit reconstruction of the continuing instrument calibration verification (e.g. test method, instrument, analysis date, each analyte name, concentration and response, calibration curve, or unique equations or coefficients used to convert instrument responses into concentrations).
- Criteria for the acceptance of a continuing instrument calibration verification are established (e.g., relative percent difference) and documented in the relevant SOPs.
- If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions will be performed. If routine corrective action procedures fail to produce a second consecutive (immediate)

calibration verification within acceptance criteria, a new initial instrument calibration will be performed. Sample data associated with an unacceptable calibration verification may be reported as qualified data only by client agreement. Chester LabNet makes no variances for out of control data reporting without client agreement.

Continuing Calibration Blank (CCB) - analyzed immediately after the CCVs, and again
after the last sample of the analytical run; for greater than ten samples, must also be
analyzed at a frequency of 10 percent or every two hours, whichever is more frequent;
where the absolute value of the result is above detection limits, terminate analysis,
correct problem, recalibrate if necessary, and reanalyze any samples affected by the
recalibration (usually, previous ten).

The following elements of continuing calibration blank analysis are essential, with minor variances for some analyses being noted in the appropriate SOPs:

- The details of the continuing instrument calibration blank procedure, calculations and associated statistics are included in the test method SOP.
- A continuing instrument calibration blank will be analyzed at the beginning and end of each analytical batch. The concentrations of the calibration blank must be less than the detection or quantitation limit of the instrument.
- Sufficient raw data records are retained to permit reconstruction of the continuing instrument calibration blank concentration (e.g. test method, instrument, analysis date, each analyte name, concentration and response, calibration curve, or unique equations or coefficients used to convert instrument responses into concentrations);
- Criteria for the acceptance of a continuing instrument calibration blanks are documented in the relevant SOPs.
- o If the continuing instrument calibration blank results obtained are outside established acceptance criteria, corrective actions will be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration blank within acceptance criteria, a new initial instrument calibration will be performed. Sample data associated with an unacceptable calibration blank may be reported as qualified data only if either the client agrees that this is acceptable, or if the technical director for that department approves a variance, which shall be documented in the data file.

Where reanalysis is not possible, the client will be notified, and if reported, the data will
be annotated in such fashion as to make clear the failure. Annotations of this nature
usually appear in the case narrative of the data report.

8.2.2 Sample Preparation and Analysis

The accuracy and precision of the overall analysis method are monitored by the analysis of QC check samples. The frequency of analysis of method QC checks is related to the number of samples prepared at the same time. This number excludes the instrument performance QC checks, but does include field samples, field blanks and replicates, and any laboratory control samples. Method QC check sample specifications and preparation procedures and the calculation methods for QC statistics are exactly as given in U.S. EPA (1994) and are outlined in Section 2.0. More detailed descriptions of the statistics can be found in the SOP for that analytical technique (see Appendix B). The specific QC checks used are as follows:

- Preparation (Reagent) Blank analyze one per preparation batch or at a minimum frequency of 5 percent; for all results above detection limits, consult with the QA/QC Coordinator; corrective actions may run from adjusting sample results for positive bias to redigesting samples and reanalyzing.
- Method (Material or Matrix) Blank (This blank is used in tandem with a preparation blank any time a sample is collected on a solid matrix (e.g., filter or sorbent tube)) analyze one per preparation batch or at a minimum frequency of 5 percent; for all results above detection limits, consult with the QA/QC Coordinator; corrective actions may run from adjusting sample results for positive bias to discarding the material, using new and reanalyzing.
- Duplicates analyze one per preparation batch or at a minimum frequency of 5 percent;
 QC statistic is the relative percent difference (Section 2.2); no immediate corrective actions, however, continual control limit exceedances warrant evaluation of the protocol, instrument, and analyst performance.
- Predigestion Spikes analyze one per preparation batch or at a frequency of 5 percent;
 QC statistic is the percent recovery (Section 2.2); no immediate corrective actions,
 however, continual control limit exceedances warrant evaluation of the protocol,
 instrument, and analyst performance.

- Laboratory Control Standards analyze one per preparation batch or at a frequency of 5
 percent; QC statistic is the percent recovery (Section 2.2); corrective actions include but
 are not limited to redigestion and reanalysis of the sample batch, however, for analysis
 where matrix interference may be involved (e.g., filters, sorbent materials etc) contact the
 QA/QC Coordinator.
- Analytical Spikes for analyses where insufficient sample is present to allow for a
 predigestion spike, analyze once per preparation batch or at a minimum frequency of 5
 percent; QC statistic is the percent recovery; no immediate corrective actions, however,
 continual control limit exceedances warrant evaluation of the protocol, instrument, and
 analyst performance.
- For sample results above the highest calibration standard, the sample will be diluted and reanalyzed where possible. This holds true for all calibration scenarios, including the 2 point calibration utilized for ICP analysis. This is to avoid the need for running linear range studies. Refer to appropriate SOPs for further details.

8.2.3 Quality Control Documentation

The Chester LabNet LIMS tabulates QC statistics and provides a hard copy of the results for client reports and for the in-house central files. The QA/QC Coordinator reviews all of the QC generated by the general chemistry laboratory. The technical directors of other areas (XRF and Weighroom) are responsible for QC oversight of data produced within their department. An example LIMS-generated QC summary sheet is provided in Figure 8.1.

8.2.4 Departures from Documented Policies and Procedures

While much of the work performed by Chester LabNet is routine analysis following published methods, occasions regularly arise when either the sample or the method do not fit well into the current documented protocols either due to sample matrix, sample collection variances, method selection by the client or any number of other reasons.

Chester LabNet does not depart from documented policies and procedures unless absolutely necessary and never without client involvement. In some cases, the client will contact the laboratory in advance with directives for deviations from methods or procedures. In other cases, the client will be contacted either by management or by the project manager to verify that necessary departures from standard operating procedures or promulgated methods are acceptable, and the laboratory will explain to the best of its ability why the deviations are needed (e.g. some CFR methods are antiquated, and some variation from the published method will, out of necessity, be required). In all cases, client agreement to the variances is required before proceeding. For long term projects, this agreement is only obtained at the beginning of the project. Subsequent sample lots will be treated following the same procedure as the initial sample lot.

Management may only grant exceptional departures from documented policies and procedures when the client has been notified and agrees to the changes being proposed.

8.2.5 Control and Technical Corrective Actions of Nonconforming Testing

Responsibility for noting discrepancies and nonconformances is born by all staff members. The first step in addressing any discrepancy or nonconformity is an evaluation of the significance of the nonconformity. Should a nonconforming item be insignificant to the final data production (e.g. typographical errors), corrections are made by the appropriate personnel, and those changes are dated and initialed. If the nonconformance is deemed significant to the final data, the appropriate technical director will investigate the cause of the nonconformance and recommend a course for corrective action. In the vast majority of cases, corrective actions are simple, are carried out immediately, and the data is brought back into conformance prior to being released to the client. The technical directors for each department are responsible for authorizing the resumption of work or the release of data to the client.

Variances noted during log in (e.g. sample ID errors, sample matrix errors, broken or contaminated samples etc) are brought to the attention of the appropriate manager or project

manager. These discrepancies are discussed with the client, and work will not proceed until the client has agreed upon a course of action. Documentation of the agreement may be as limited as notations on the chain of custody form or as formal as a client contact form, depending on the nature of the issue.

Due to the wide variety of methodologies utilized at Chester LabNet and the wide range of sampling techniques employed by clients, discrepancies occurring at the sample preparation level or the analysis level span a wide range of issues. If the discrepancy is capable of being handled by the analyst, it is the responsibility of the analyst to correct or otherwise resolve it (e.g. rerunning samples, recalibrating the instrument, etc). In cases where the discrepancy can not be resolved by the analyst, the discrepancy shall be reported to the appropriate manager or technical director for further action. Depending on the nature of the discrepancy, corrective actions may be as simple as notations in the case narrative or as complex as method development. In all cases, the client is notified of the discrepancy, and the client is responsible for determining how to proceed. Data will not be released to the client until such time as corrective actions have been discussed, and, where applicable, the client has agreed that the data is acceptable. Any data reported to the client with out-of-control QC shall be noted in the case narrative, along with any other pertinent information as to the suspected causes of the QC failures where known.

Should nonconformances be discovered after the release of data to the client, the client is immediately contacted and informed of the nature of the nonconformance. The source of the nonconformance is identified, corrected where possible, and the corrected data resent to the client.

8.2.6 Formal Corrective Actions

Should the occurrence of nonconforming data indicate a systemic problem with methods or procedures, the laboratory will investigate to determine the root cause(s) of the problem. The laboratory will select and implement the action most likely to prevent recurrence of the problem. Corrective actions may be as small as adding an extra layer of data review or as large as a complete modification of a technique. All changes will be documented in their associated SOPs. The appropriate technical director will monitor the results of the changes, and ensure that the changes are being followed and have resolved the issue. For greater detail, refer to SOP QA-002, "Laboratory Data and Report Validation".

QA/QC Report

Client Name: Project Number:

Analytical Technique: ICP Sample Description: 8x10 QMA

Report Number:

Calibration QC

	Sample	Standard	Measured	Percent
Analyte	ID	Conc. mg/L	Conc. mg/L	Recovery
Pb	ICV	1.00	0.95	95.4
Pb	CCV	1.00	1.01	101.0

Blank Data

	Sample	Measured	MDL
Analyte	ID	Conc. mg/L	Conc. mg/L
Pb	ICB	< MDL	0.80
Pb	Prep_Blk	< MDL	0.80
Pb	Meth_Blk	< MDL	0.80
Pb	CCB	< MDL	0.80

Duplicate Data

	Sample	Standard	Measured	Percent	
Analyte	ID	Conc. mg/L	Conc. mg/L	Recovery	
Pb	01-Q357	< 0.08	< 0.08	N/C #	

Laboratory Control Sample/Matrix Spike Analysis

	Sample	Standard	Spike	Spike	Percent
Analyte	ID	Conc. mg/L	Conc. mg/L	Amount mg/L	Recovery
Pb	LCS	< 0.08	0.908	1.00	90.8
Pb	01-Q359	< 0.08	0.883	1.00	88.3 #

QA/QC Limits

Continuing Calibration: 10% LCS: 20% Duplicates: 20% RPD Spikes: 25%

 $RPD = \{(sample-duplicate)/[(sample+duplicate)/2]\}x100$

N/C: RPD is not calculated when sample or duplicate is below detection limit
#: per EPA CLP protocol, control limits do not apply if sample and/or
duplicate concentration is less than 5x the detection limit

Figure 8.1 Example LIMS-Generated QC Summary Sheet

8.2.7 Preventative Action

All employees are responsible for identifying possible opportunities for improvement where they notice such opportunities. If preventative action is deemed necessary, the technical director for the appropriate department will be notified, changes implemented, and changes made to the applicable SOP. Where pertinent, application of controls will be established to ensure that the preventative action is effective and working as intended.

8.3 Management of Complaints from Clients

While inquisitions into data are not uncommon, actual complaints by clients are quite rare. In either case, once contacted by the client, the project manager notifies the appropriate technical director or the QA/QC director of the nature of the issue. That person locates the original data report and investigates the client's concern.

8.3.1 Errors Made by the Laboratory

While rare, errors do occasionally occur by the laboratory staff. If the issue at hand is the result of an error made by the laboratory (e.g. miscalculation, transposed numbers, decimal point errors, incorrect sample IDs, etc.), the laboratory will correct the error and issue a new corrected report to the client. In some cases, the client may request that the samples be reanalyzed. Where possible, this is performed.

8.3.2 Issues Resulting from Sample Characteristics

Air Quality sampling is not a simple matter. Issues may arise over which the laboratory has little or no control (e.g. filter deposits not adhering to the filter, stack (source) samples having interfering analytes, impinger solutions with large quantities of particulate matter etc). In these cases, and where possible the client is notified prior to work being performed, and client agreement as to how to reconcile the matter is noted in the report. Where the client had been previously notified of such issues, the client complaints are referred back to the client's original statements. In cases where the issue could not be detected until after analysis (such as interfering compounds), the client will be notified prior to receiving the data. The laboratory will explain the cause of the problem to the client, as well as what, if any, other courses of action may be taken to resolve the issue. In some cases, the client may request that the samples be reanalyzed or analyzed following a different method. Where possible, this is performed.

8.3.3 Unethical or Illegal Requests by Clients

On rare occasions, client complaints take the form of the laboratory refusing to commit unethical or illegal actions. Clients which request the laboratory to perform such actions are declined their request and an explanation given as to why the request is declined. Such requests have taken the form of asking the laboratory to analyze Sample A, but report the results as Sample B due to the loss of Sample B during shipping, or requesting that reanalysis be performed until the number desired by the client is obtained. Chester LabNet does not and will not report false data to any client. Samples from clients who persist in making such requests shall be refused in the future. Chester LabNet will not knowingly, directly or indirectly, participate in fraudulent activity. Any indications that the client may be using Chester LabNet's data in a fraudulent manner are documented in the case narrative and/or other areas of the data report.

8.3.4 Billing Complaints

Client complaints regarding billing errors are directed to the president, who also performs all accounting functions for the company. The president will investigate the billing in question. Where errors are found he will issue a new statement and rectify the financial records to show the correct billing amount.

8.3.5 Media and Supplies Complaints

Complaints regarding sampling media are referred to either the project manager or the Technical Director responsible for that particular media. In instances where the incorrect media was shipped to a client, the error will be corrected and appropriate media sent in a timely fashion to the client. It is Chester LabNet's policy that media, once sent to a client, can not be returned unused as the laboratory can not vouch for the integrity of the media once outside of its control. Complaints about the inability to return unused media are explained by this policy, with which most clients agree once they understand the logic behind it.

8.3.6 Documentation of Complaints

All customer inquiries or complaints are recorded on a "Customer Inquiry/Complaint" form. This form is completed by the employee handling the complaint, and eventually stored in the Customer Inquiry/Complaints log kept by the President. This log is reviewed annually, during the management review cycle.

8.4 Traceability of Measurements

All equipment used for environmental analysis, including equipment for subsidiary measurements (e.g. environmental conditions) having a significant effect on the accuracy or validity of the result of the environmental test will be calibrated before being put into service and verified on a continuing basis. The appropriate laboratory SOPs specify the procedure for the calibration of the equipment. This includes balances, thermometers, and control standards. The appropriate SOPs describe the process for selecting, using, calibrating, checking, controlling and maintaining measurement standards, reference materials used as measurement standards, and measuring and test equipment used to perform environmental tests and calibrations.

9.0 Data Reduction, Review/Validation, and Reporting

9.1 Data Reduction

For gravimetric analyses, the only data reduction is the subtraction of the filter tare weight from the gross weight, which is done automatically by the dedicated computer interfaced with the analytical balance. There is no further mathematical manipulation because the resultant net weight is already on a per-filter basis.

For all other analyses, data reduction is accomplished by the calculating methods specified in the analytical protocols listed in Tables 4.1 though 4.3, by the calculating methods specified in the appropriate SOP, automatically by the software controlling each instrument, or by the LIMS. The most common data reduction calculations involve analysis results, sample preparation conditions, and physical data supplied by the client. For example, in the analysis of air particulates on filters for lead, the instrument results are reported in units of μg Pb/L of extract. The sample preparation conditions may be that exactly one-fifth of the filter was extracted in 10 mL. The first data reduction step then becomes:

$$\mu$$
g Pb/filter = (μ g Pb/L) x (0.010 L/filter) x 5

The concentrations of analytes in air particulates are most often reported in units of percent net mass and $\mu g/m^3$. The final data reduction steps take information supplied both by the client (total volume of air sampled) and/or the LabNet weighroom (total mass on the filter) to calculate the result as it will be reported:

% Pb of net mass = (
$$\mu$$
g Pb/filter) x (filter/ μ g particulate) x 100

$$\mu g Pb/m^3 = (\mu g Pb/filter) x (filter/m^3 sampled)$$

The project manager will enter into the LIMS the original result in μ g/L, the extract volume in L, the total filter deposit area, and the extraction/digestion area of the filter used during the preparatory steps. The air volume will already have been entered into the LIMS at the time of sample log in if the client has provided that information. The total mass on the filter would have been automatically entered into the LIMS during the weighing procedure. The actual calculations will be performed by the LIMS itself.

9.2 Data Validation

Three levels of data validation are performed. Levels 0 and I are performed on all samples received by Chester LabNet. Level II data validation is only performed when required by contractual obligation. The purpose of data validation is to ensure that the reported data are free from transcription and calculation errors (manual or electronic), and that all quality control measures are reviewed and evaluated prior to data being reported.

9.2.1 Level 0

Level 0 validation occurs at the sample receipt and log in stage of sample analysis. Elements of Level 0 validation include:

- examining integrity of custody seals, if present
- taking the temperature of a transit temperature bottle, if present
- examining integrity of shipping bottles or containers
- examining the chain of custody (COC) form(s) for the presence of all required information and signatures
- verifying sample ID numbers against those listed on the COC form(s)
- contacting the appropriate authority upon finding irregularities, then documenting and carrying out corrective actions

For projects requiring additional documentation of the level 0 validation process, LabNet provides a written checklist covering the above steps. This checklist is filled out, signed, and dated by the Sample Custodian or designated alternate. The completed checklist is added to the project file.

9.2.2 Level I

Level I data validation begins during sample analysis and is carried out at the instrument by the analyst. This phase of level I validation involves performing and maintaining instrument calibration and assessing precision and accuracy of the data via the analysis of all of the appropriate QC checks, as discussed in Section 8.0 The analyst ensures that the QC statistics are within control limits and takes appropriate corrective actions during analysis if control limits are exceeded.

For projects requiring additional documentation of the level I validation process, LabNet provides a written analyst's checklist. This checklist is filled out, signed, and dated by the analyst. The

completed checklist is added to the project file. An example of a Level I instrument data review checklist is shown in figure 9.1.

The second phase of level I data validation is performed by the QA/QC director or Technical director for that particular department. During this phase, raw data is verified as being in control with the appropriate QC parameters, worklists are checked for accuracy against the raw data, raw data is checked for any discrepancies which may have been missed by the analyst and any corrective actions are taken to correct deficiencies prior to the data being submitted to the project manager.

The third phase of level I data validation is performed by the project manager or LIMS Administrator, who confirms all keyboard entries and electronic data entries into the LIMS, then confirms that the correct analyses have been completed on the correct samples. The project manager or LIMS administrator then reviews all of the data and QC results for a given project or report and for certain clients prepares QC summary tables and data assessments. Problem data discovered during this review are flagged.

If any analytical errors are found in any of these stages of data review, and if there is enough sample extract remaining and if holding times have not been exceeded, the preparation and/or analysis will be redone and the new results will be subjected to the same QC/validation. SOPs QA-002 and AD-007 are the most relevant to this stage of review.

9.2.3 Level II (CLP reports only)

Level II data validation is only performed for CLP style reports and is carried out by the QA/QC Coordinator and occurs after the data package has been correctly assembled. The first step is to recalculate by hand the final result for a randomly picked sample. This is accomplished by first taking the raw calibration data and recalculating the appropriate calibration statistics (i.e., slope, intercept, and correlation coefficient). Next, using the raw instrument response, the instrument concentration result is recalculated. Finally, the sample preparation data (i.e., digestate volume, filter aliquot size, etc.) are used to recalculate the final result as reported to the client. All of these steps are documented on a Sample Calculation preprinted form, which is signed and dated by the reviewer and included in the final data report. An example Sample Calculation form is provided in Figure 9.2.

The second step is to review all QC statistics and raw data for compliance to control limits, frequency of application, and correct sequences. In addition, flagging is checked as well as

reporting units, holding times, and the correct use of significant figures. Finally, corrective actions (if applied) are noted. The review is aided by following a preprinted checklist, which is signed and dated by the QA/QC Coordinator and placed in the data report. An example Data Package Review Checklist is provided in Figure 9.3. Results for all data review, verification and cross checking procedures are documented within each data package, to the extent that is required for each particular client's needs. At a very minimum, documentation shall consist of at least one person's signature or initials attesting to the performance of data review.

9.3 Data Reporting

All data are reported in commonly accepted units, or as specified by the client. Reports are reviewed by the project manager or president, who gives final approval for release. Depending upon the analysis and the requirements of the client, raw data report forms created by the instruments may be included in the final report. If an instrument raw data report is not required, it is retained in hardcopy form and on a computer file for future reference. Computer files will be kept by the laboratory for a minimum of 90 calendar days. Hardcopy files will be retained by the laboratory for a minimum of 5 calendar years. Final reports are created by the LIMS. An example final report page for the analysis of particulates on air filters is provided in Figure 9.4.

9.3.1 Electronic Data Reports

Chester LabNet provides electronic data deliverables in a variety of formats on a client specific basis. Previous e-reports have been sent as either email attachments or on a floppy disc via common courier. Files have been formatted as: CSV files, dbase files, fixed width column files, spreadsheet files, text files or proprietary client software files. Chester LabNet works closely with the client to ensure that e-reports are in a useable format.

9.3.2 Hard Copy Data Reports

9.3.2.1 Draft/Screening reports

These reports consist of the results for each sample and may be faxed to the client. All pertinent QC is performed by the laboratory, and hardcopy reports are retained on site, however they are not reported or delivered to the client. Normal use of this report format is either for immediate reporting of draft data for rush purposes, or for engineering data where the engineers are establishing a baseline or performing investigatory work. This

report format is never used for compliance purposes.

9.3.2.2 Standard reports

These reports contain all of the results for the samples and associated QC reports, plus any legal documents (chain of custody forms, telephone logs, etc.) pertaining to the samples. They also include a case narrative and a summary of the analysis performed on the samples. The QC reports are not in CLP formatting. Raw data is included only upon request.

9.3.2.3 CLP reports

These reports are frequently mislabeled "Level IV" by clients (see section 9.3.3). They include all raw data, all appropriate CLP data and forms in addition to the items contained in a standard report. This reporting format is usually only generated by client request and at an extra charge to the client.

9.3.3 Discussion of CLP ('Level IV') data reporting.

9.3.3.1 History of 'Level IV'

In 1986, the Director of the Office of Emergency and Remedial Response began using the terms Level I, Level III, Level IV and Level V as jargon for various levels of Data Quality. These levels were defined as follows (quoted from EPA 540/G 87/003A, Data Quality Objectives For Remedial Response Activities, March 1987)

"Level I-Field screening. This level is characterized by the use of portable instruments that can provide real-time data to assist in the optimization of sampling point locations and for health and safety support. Data can be generated regarding the presence or absence of certain contaminants (especially volatiles) at sampling locations.

Level II-Field analysis. This level is characterized by the use of portable analytical instruments that can be used on-site, or in mobile laboratories stationed near a site (close-support labs). Depending upon the types of contaminants, sample matrix, and personnel skills, qualitative and quantitative data can be obtained.

CHESTER LABNET-PORTLAND INSTRUMENTAL QC CHECKLIST FOR ICP, GFAA, CVAA

Method		Project	
Date		Batch ID	
Analyst		Analytes	
	YES NO	CORRECTIVE ACTION	IS/
PRELIMINARY QC CHECKS		COMMENTS	
COMPUTER DATE/TIME CHECKED			
PRIMARY, ICV, CCV STD ID NUMBERS NOTED			
PRIMARY STD WITHIN EXPIRATION DATE			
CORRECT # STDS PREPARED			
CORRECT CONC. RANGE FOR STDS			
ICV PRIMARY STD DIFFERENT FROM CALIBRATION STD			
CCV PRIMARY STD DIFFERENT FROM CALIBRATION STD			
CALIBRATION QC CHECKS			
CORRELATION COEFF. WITHIN CONTROL LIMITS			
ICB RESULTS BELOW DET. LIMITS			
ICV % RECOVERY WITHIN CONTROL LIMITS			
CCB-1 BELOW DET. LIMITS			
CCV-1 % RECOVERY WITHIN CONTROL LIMITS		<u> </u>	
NO MORE THAN TEN ANALYSES BETWEEN CC'S		<u> </u>	
CCB-2 BELOW DET. LIMITS			
CCV-2 % RECOVERY WITHIN CONTROL LIMITS		<u> </u>	
NO MORE THAN TEN ANALYSES BETWEEN CC'S			
CCB-3 BELOW DET. LIMITS			
CCV-3 % RECOVERY WITHIN CONTROL LIMITS			
NO MORE THAN TEN ANALYSES BETWEEN CC'S			
CCB-4 BELOW DET. LIMITS			
CCV-4 % RECOVERY WITHIN CONTROL LIMITS			
NO ANALYSES AFTER FINAL CC'S			
DIGESTION/ANALYSIS QC CHECKS			
NO MORE THAN 20 SAMPLES FOR THESE QC CHECKS			
ALL RESULTS WITHIN INSTRUMENT CALIBRATION RANGE			
PREPARATION BLANK BELOW DET. LIMITS			
LCS % RECOVERY WITHIN CONTROL LIMITS			
DUPLICATE RPD WITHIN CONTROL LIMITS			
IF NO PASS, DO CONTROL LIMITS APPLY?			
IF CONTROL LIMITS APPLY, AFFECTED DATA FLAGGED?			
SPIKE STD ID # NOTED & DIFFERENT FROM CAL STD			
MATRIX SPIKE % RECOVERY WITHIN CONTROL LIMITS			
IF NO PASS, DO CONTROL LIMITS APPLY?			
IF CONTROL LIMITS APPLY, AFFECTED DATA FLAGGED?			
SPECIAL GFAA DIGESTION/ANALYSIS QC CHECKS			
GFAA ANALYSES ALL IN DUPLICATE			
DUPLICATE RSD'S WITHIN CONTROL LIMITS			
IF NO PASS, DO CONTROL LIMITS APPLY?			
IF CONTROL LIMITS APPLY, AFFECTED DATA FLAGGED?			
FINAL QC CHECKS			
ALL LABORATORY NOTEBOOKS COUNTERSIGNED & DATED?			

Figure 9.1 Example Instrumental QC Checklist

CHESTER LABNET-PORTLAND SAMPLE CALCULATION

RMA Contract No. SDG # Analytical Method			- - -		Reviewer Date	
Analyte	1		=			
CALIBRATION						
		Conc.	Instrument Response	Page In Data Package		
	Blank					
	Std #1					
	Std #2					
	Std #3					
	Std #4					
	Std #5]	
	ſ		Page In		7	
		Report	Data Package	Check		
	Slope	· · · · · · · · · · · · · · · · · · ·			1	
	Intercept				1	
	r				1	
	<u> </u>				-	
SAMPLE RESULT C	ALCULATIO	N				
Sample						
Raw Data				•		
	ize/Dilution Da nal Result Paç					
Reported	Result			µg/filter		
	n Check Resu	ult		μg/filter		
Calculation Check						
X = Y-b	=			_		
m = 1-b				. =		
Metals MDLs (ug/filt	er):					
	,.					

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Figure 9.2 Example Sample Calculation Form

CHESTER LABNET-PORTLAND DATA PACKAGE REVIEW CHECKLIST

Contract # SDG # Analyte/Test Code	Reviewed by Review Date Analytical Method			
	CI ICP	HECKED ¹	? CV	CORRECTIVE ACTIONS/COMMENTS
PRELIMINARY				
HOLDING TIMES MET FORM I: SIGN. FIGURES		\vdash	H	
UNITS			H	
FLAGS				-
QC STATISTICS				
ICV/CCV'S				
ICB/CCB'S				
LCS				
METHOD BLANK		\vdash	\mathbf{H}	
MATRIX SPIKE RAW DATA				
ANALYSES IDENTIFIED				
CALIBRATION			\Box	_
DATA WITHIN CAL. RANGE				
RUN SEQUENCE CORRECT				
GFAA IN DUPLICATE				
CALCULATIONS				
CALIBRATION			\square	
SAMPLE FINAL RESULT	Ш	Ш	Ш	-

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Figure 9.3 Example Data Package Review Checklist

Level III-Laboratory analysis using methods other than the CLP RAS. This level is used primarily in support of engineering studies using standard EPA approved procedures. Some procedures may be equivalent to CLP RAS, without the CLP requirements for documentation.

Level IV-CLP Routine Analytical Services (RAS). This level is characterized by rigorous QA/QC protocols and documentation and provides qualitative and quantitative analytical data. Some regions have obtained similar support via their own regional laboratories, university laboratories, or other commercial laboratories.

Level V-Non-standard methods. Analyses that may require method modification and/or development. <u>CLP Special Analytical Services (SAS) are considered Level V."</u>

In September 1993, Henry Longest, then Director of the Office of Emergency and Remedial Response, issued a memo (EPA Directive No. 9355.9-02) doing away with Levels I-V as a means of describing various levels of usability of data. Quoting from this Directive:

"....The major changes from the 1987 DQO guidance are (1) that the qualitative approach has been supplemented with quantitative aspects, (2) that the guidance is directed toward the entire Superfund Process, not just the remedial program, (3) that the five analytical levels in the 1987 guidance (as well as the three quality assurance objectives in the QA/QC removal guidance and the three data usability categories (DUCs) in the site inspection guidance) have been replaced by two data categories.

...The Interim Final Guidance on Data Quality Objectives Process for Superfund supersedes the 1987 guidance in total, including the five analytical levels. The Interim Final Guidance also replaces the sections of the QA/QC removal guidance which relate to the three quality assurance objectives and the sections in the site inspection guidance which relate to the three data usability categories."

The two data categories referenced in this quote are "definitive" and "screening" data. For laboratory data review, the following elements are listed in Guidance For Data Quality Assessment (EPA QA/G-9, Final, July 2000), section 2.1.1:

The first activity in conducting a preliminary data review is to review any relevant QA reports that describe the data collection and reporting process as it actually was implemented. These QA reports provide valuable information about potential problems or anomalies in the data set. Specific items that may be helpful include:

- Data validation reports that document the sample collection, handling, analysis, data reduction, and reporting procedures used;
- Quality control reports from laboratories or field stations that document measurement system performance, including data from check samples, split samples, spiked samples, or any other internal QC measures; and
- Technical systems reviews, performance evaluation audits, and audits of data quality, including data from performance evaluation samples.

When reviewing QA reports, particular attention should be paid to information that can be used to check assumptions made in the DQO Process. Of great importance are apparent anomalies in the recorded data, missing values, deviations from standard operating procedures, and the use of nonstandard data collection methodologies.

9.3.3.2 **Summary**

The new data quality objectives of the EPA are now significantly more flexible than they had been under the "level" system. The QA/QC requirements now vary from one sampling site to another, based on the specific needs of that sampling site and analytical methodologies. QA/QC requirements for defensible data are now judged not on a set of predefined criteria which may or may not apply to the specific site/analytes being studied, but vary from site to site, project to project, and are judged based on their applicability to the methods employed in all stages of site remediation and engineering.

9.3.3.3 Chester LabNet "CLP" reports

Contract Laboratory forms (CLP) were initially designed for reporting metals analysis of waters and solids. These forms can be utilized to report metals analysis in air quality samples, as well as reporting nutrient/acid rain components once the forms have been modified to accurately reflect the analyte(s), matrix, and appropriate units.

Given that every client, site, and method vary in their QA/QC needs, Chester LabNet will work in tandem with the client to ensure any CLP formatted reports meet that client's particular need for that project.

9.4 Client Confidentiality and Data Ownership

All data produced by Chester LabNet is the property of the client. As such, Chester LabNet will not and does not release data to any other person, agency or business without the prior verbal or written consent of the client. Verbal consent is documented and maintained in the data report.

In cases where data is subpoenaed, Chester LabNet will contact the laboratory's lawyer prior to submitting data. In these rare instances, only the data directly mentioned in the subpoena shall be released to the subpoenaing authority. Data which may be related to the subpoena but was generated for a different client must be subpoenaed independently. Any situations arising involving legal action shall be brought to the attention of the laboratory president, and shall

involve Chester LabNet's representing lawyer to ensure the subpoena is correct, pertinent, legally viable and that any actions taken by Chester LabNet in releasing data are legally defensible.

Client: XXXXXXXXX

Project Number: X####

Lab ID: 01-Q357
Client ID: FL2-110701
Sample Date: 11/ 7/01
Mass: 60700

Mass: 62700. +- 500. ug

Deposit Area: 406. cm2 Comments: 011170E-01A

Analyte g/filter percent

ICP

Pb 40.1 0.063

Lab ID: 01-Q359 Client ID: RR-110701 Mass: 56200. +- 500. ug

Deposit Area: 406. cm2 Comments: 011170E-02A

Analyte g/filter percent

ICP

< 39.1 Pb < 0.070

Lab ID: 01-Q360
Client ID: RR-111201
Sample Date: 11/12/01
Mass: 39400. +- 500. ug

Deposit Area: 406. cm2 Comments: 0111302E-02A

Analyte g/filter percent

ICP

< 39.1 < 0.099 Pb

Figure 9.4 Example Final Data Report Page for standard data packages.

10.0 Performance Evaluations, System Audits and Demonstration of Capability

10.1 Introduction:

The QA/QC Coordinator, with the assistance of the Project Manager and any applicable Technical Directors, will be responsible for ensuring that all LabNet personnel are following QA/QC requirements and sound scientific practices. This will be accomplished, in part, by the performance of periodic audits of program activities. An audit is a formal examination of all of the contractual provisions and relevant components of this or any other QA Plan to verify that the components are being faithfully carried out by the laboratory at all levels. Chester LabNet will afford clients (or their representatives) cooperation to clarify the client's request and monitor the laboratory's performance in relation to the work performed.

These audits are the principle means of review to determine compliance with established QA program goals and procedures. It is highly important that personnel who participate in audit activities understand that the audit is a means of documenting scientific procedures and is not an attack on personal integrity.

10.2 Ongoing Management Assessment

The management assessment that the laboratory is operating within its QC program occurs with the review of all data reports by the President. The President's review signifies verification of adherence to the laboratory QC system, including assessment of the quality of the analytical data and the meeting of the QC requirements of the QA Management Plan and the Laboratory QC Plan.

10.3 External Audits

10.3.1 Performance Audits

Historically, Chester LabNet participated in two annual external performance audits: the U.S. EPA WS study for drinking water and the U.S. EPA WP study for the NPDES program. Both of these programs involved the analysis of blind performance evaluation samples for selected inorganic constituents. In addition, the WS study entailed the submission and approval of a laboratory QA plan and a site inspection by the state of Oregon.

These programs were discontinued with the advent of the National Environmental Laboratory Approval Program (NELAP). Unfortunately, NELAP does not have Performance Evaluation standards for the methods employed at Chester LabNet. Due to a lack of standards for the methods utilized at Chester LabNet, the laboratory can not participate in a proficiency test program through the NELAC program.

For other contracts and/or accreditations, client supplied proficiency test sample results are maintained in the same manner as any other data report would be, with the exception that these reports are not given report numbers, rather are kept with the contract/accreditation documentation maintained by the laboratory president.

10.3.2 Technical Systems Audits

Chester LabNet has undergone several technical systems audits on a project-specific basis for private and governmental clients. These audits have included a site visit to verify the existence of and to evaluate laboratory physical plant, scientific equipment, supplies, personnel, training, and operating procedures. Also included have been the analyses of blind performance evaluation samples. Recent technical system audits have included the following:

- U.S. EPA for the Special Analytical Services branch of the Contract Laboratory Program
- Bechtel Environmental for the Eastern Michaud Flats CERCLA/RCRA Remediation
- URS Consultants for the Vertac Superfund Site CERCLA Remediation
- Engineering Science for the Laskin Poplar Superfund Site CERCLA Remediation
- Fluor-Daniel for the Big D Campground Superfund Site CERCLA Remediation.
- URS Group Rocky Mountain Arsenal Site Remediation.
- ICF Consulting via Bill Osluand and Associates for the Mobile County Air Quality Task Force
- Amec

All CARs, audits results and audit responses are retained in client or accreditation specific files maintained by the laboratory president.

10.4 Internal Audits

An Internal audit of all systems is performed annually by the Quality Assurance Officer. The audit consists of seven sections, audited separately over the course of one year: Management Review, Quality Systems Audit, Customer Services, XRF, Inorganics Laboratory, Technical Overview, and Weighroom Audit (gravimetry).

The audits are structured following the most current ORELAP Chapter 5 checklist available online at the time the audit forms were created. Each area audit is performed using a checklist specific to that area of the laboratory functions, and copied directly from the ORELAP checklist. Thus the areas audited are being held accountable to the ORELAP (and therefore NELAP) standards.

After each audit and audit checklist has been completed, and audit summary is completed. Deficiencies are noted on an audit deficiency form. The same form also contains sections to be completed for the proposed corrective action, date by which the corrective action will be carried out, follow-up to ensure corrective action was completed and an area for the QAO to sign off that the deficiency has been fully resolved. If the deficiency casts doubt on the validity of any test results, clients will be notified as soon as practicably (not more than 5 business days from the day of discovery), either by phone or email.

At the end of the annual audit cycle, after all seven sections have been through the audit/review and all deficiencies resolved, an annual audit summary report will be written. This report will summarize the findings, corrective actions, follow-up procedures and any other items of note found during the annual audit.

Refer to Section 3.0 of this document for insuring that any evidence of inappropriate actions or vulnerabilities related to data integrity are monitored on an ongoing basis.

10.5 Managerial Reviews

Managerial reviews are part of the annual internal audit/review process described above.

10.6 Performance Evaluation samples

Clients will occasionally submit blind Performance Evaluation (PE) Samples for various methods and matrices. When possible, Chester LabNet requests that the laboratory be informed of their performance. Historically, Chester LabNet's results on such PE samples have been well within control of the limits set by the client or agency requesting the PE sample. Requests for results of recent PE samples run for other clients shall be met by the laboratory to the extent possible without violating any client confidentiality issues.

10.7 Demonstration and Documentation of Capability

Prior to the institution of any new method that has not been in use by the laboratory prior to July 1999, demonstration of capability will be performed. Demonstration of capability will be considered adequate when all QA/QC parameters for that method, which the laboratory is capable of meeting, can be met by the analyst performing the work (e.g. methods requiring gaseous spiking of sorbent tubes with analytes of interest is not possible, however, liquid spiking of the sorbent material is possible and will be used in lieu of gaseous spikes). Continuing demonstration of method performance will take the form of continued meeting of established QA/QC criteria. Demonstrations of capability will be completed each time there is a change in instrument type, personnel or method.

For procedures which have been in use prior to July 1999, demonstration of capability will consist of continuing to meet established QA/QC protocols, and shall be documented within each data package showing the analyst's ability to meet said protocols. In-house run logs, where applicable, shall serve as proof of the amount of time an analyst has been running a given method. For infrequently run methods, data packages shall serve as proof of the amount of time an analyst has been running a given method. Due to the wide variety and extreme specificity of certain air quality methods, some methods may be run only once every three years or less, making ongoing certification for all methods an undue burden on the laboratory. In such cases, the passing of QC parameters shall be taken as demonstration of proficiency.

11.0 Document Control and Records

11.1 Program Documents: Process, Approval and Distribution

Currently, Chester LabNet has two program documents: Laboratory Quality Assurance Management Plan (QAMP) and the Chemical Hygiene Plan (CHP). All program documents are written and polished to a draft condition by the QA/QC Coordinator prior to being submitted to the company President as a draft version for review. The President makes comments on the draft version and resubmits it to the QA/QC Coordinator for revision. After making the requested revisions, program documents are circulated to the rest of the staff for comments. Once all comments have been addressed, the final copy is read and signed by all Chester LabNet personnel. The documents are stored in three ring binders in the main office area of the premises, and are reviewed annually by all original signatories where possible, or their replacements.

11.2 Document Control

All original documents are kept in white three ring binders in the main office area. Original finalized copies of documents or three ring binders containing documents may not be removed from the premises under any circumstances. Photocopies, electronic copies, and/or draft copies of documents may only be removed from the premises with the approval of the QA/QC Coordinator.

Due to the small size of Chester LabNet, CLN has no formal document control system, nor does CLN have an SOP describing document control within the company. As no copies of documents are permissible, the need to trace dispersed copies is null and void. SOP AD-003 does include a thorough description of the processes CLN uses to govern document generation, control and archiving of old documents. The QA/QC Officer maintains a master list of all in-house written documents on the laboratory computer. Technical Directors and analysts are responsible for maintaining control of instrument specific manuals and literature.

All CLN employees have access to the original documents at all times. All original documents are stored in a bookcase in the main office area. All CLN documents are reviewed on an annual basis by the QA/QC Coordinator, the appropriate technical director or an alternate designated by them, and the employee(s) performing the procedure on a regular basis. The QA/QC Coordinator is also responsible for the preparation, approval and issuance of new documents.

Copies are not allowed to be made, except for submission to the client for the purposes of meeting contractual or proposal obligations. Any photocopy must be approved by either the Lead Project

Chester LabNet QA Management Plan

Manager or the QA/QC Coordinator. No copies of client specific documents (QAMP or any SOP) will be submitted to any other client. All original SOPs or CLN documents must be signed on their Cover Page or Review History Page by at least two CLN employees in <u>BLUE</u> ink. Any copy of a document will be clearly stamped with the word "copy" on the cover page, and can be distinguished from the original by a lack of signatures in blue ink.

11.3 Standard Operating Procedures (SOPs)

Original, signed SOPs are kept in several 3-ring binders in the main office area of the laboratory. The retired versions are stapled along the right margin and retained in the archived SOPs file drawer. The production of new SOPs and the revision of existing SOPs are under the supervision and control of the QA/QC Coordinator. Each SOP must be approved, signed, and dated a minimum of two people: the author and the person most familiar with the procedure. In cases where the author is the person most familiar with the technique, a second person with or without the same degree of technical knowledge shall read and sign the SOP. Within the laboratory, original copies are always used as references. Copies are only made for submission to outside authorities, and only on specific contractual request. Any copies of SOPs (e.g., to be used for submission materials for new projects or for proposals) must be stamped "COPY" in red ink across the title page. All SOPs are reviewed annually, and a master list of SOPs is maintained electronically by the QA/QC officer to ensure annual review.

11.4 QC Guidance Manuals

Originals of the general laboratory QA Plan, project-specific QA Plans, and the general laboratory Chemical Hygiene Plan are kept along with the original SOPs in the main office area of the laboratory. The production of new QC guidance manuals and the revision of existing QC guidance manuals is under the supervision and control of the QA/QC Coordinator. Each QC guidance manual must be signed and dated by the author. In addition, QA Plans must be read and signed and dated by all affected laboratory personnel. This process is conducted annually for the general laboratory QA Plan and for all project-specific QA Plans where an annual review is required. The date of issue is clearly marked on the title page, and the total number of pages is clearly marked at the top of each page, beside the specific page number.

11.5 Laboratory Notebooks

Bound laboratory notebooks are assigned numbers and dispensed by the QA/QC Coordinator or Lead Project Manager, who maintains a bound book containing the dispensed logbook number, date of origination, use, and date the logbook is retired. The master logbook tracking book is kept in the main office area of the laboratory. Filled logbooks are decommissioned by the QA/QC Coordinator. Their decommission date is noted in the same bound book, and the decommissioned logbooks are kept in a series of labeled banker's boxes in the file closet.

11.6 Document Production and Maintenance

All internal documents have a Cover Page and a Review History Page. The Review History Page tabulates the changes made over time to the document. Any major changes to the document content will be noted in this table (see Review History Page of this document). Minor changes can be noted in the original document in NON-BLACK ink, as long as those changes are dated and initialed by the person making the change. Upon the introduction of a newly revised document, the newly retired version is stapled along the right margin and retained in the archived SOPs file drawer.

The production of new documents and the revision of existing documents is under the supervision and control of the QA/QC Coordinator. Each document must be approved, signed, and dated by the author, QA/QC Coordinator (usually the same person) and at least one other CLN employee. Any copies of documents (e.g., to be used for submission materials for new projects or for proposals) must be stamped "COPY" in red ink across the title page.

All documents will be reviewed annually by the QA/QC Coordinator for currency, accuracy and clarity. Any revisions will be noted in the Review History table at the front of the document. If no changes are needed, the review will be documented by signing and dating the annual review line of the Review History page.

All documents are maintained on the QA/QC Coordinator's computer. Changes to documents are performed only by the QA/QC coordinator, or, rarely, by the president. Handwritten corrections may be made to the original hardcopy by any employee, as long as that change is dated and initialed. The review history of each document notes changes made to the document, and the name of the person making the changes. Due largely to the undesirable nature of document maintenance, as well as to the personal integrity of the employees, employees other than the QA/QC officer do not make changes to the electronic copy of the document, therefore stringent control of access to the electronic copies of the documents is not undertaken.

11.7 Analytical Record Keeping

Analytical records associated with analysis are retained in varying formats and locations such as to create a documentation trail sufficient to create a historical account of the analysis of any given sample. These records and their locations are listed in the table below:

Record	<u>Location</u>
Client/Laboratory Sample ID	LIMS and final data report
Date/time of analysis	Raw data (final data report)
Instrument ID	Header of the instrumental printout or data sheets
	(final data report)
Instrument operating conditions	Instrumental method is usually noted in the header
	of the instrumental printout or is hand written on the
	raw data (final report)
Analysis type	Final data report (data sheets or case narrative)
Manual calculations	Raw data (final data report)
Analyst's initials	Raw data (final data report)
Sample Preparation Logs	Raw data (final data report)
Sample Analysis	Final data report
Standard and reagent origin, receipt, preparation	Prep logs, standard logs, SOPs (for reagents
and use;	needing preparation immediately prior to use),
	Ordering and Receipt files
Calibration Criteria, frequency and acceptance	Appropriate SOPs
criteria	
Data and statistical calculations, review,	Final data report
confirmation, interpretation, assessment and	
reporting conventions	
Quality control protocols and assessment	Protocols contained in appropriate instrumental
	SOPs. Assessment found in raw data (final report)
Method performance criteria	Appropriate SOPs

11.8 Control of Data Reports

SOPs AD-007 and AD-008 document the production of data reports. Each data report will have all associated hard copy documents necessary for the historical reconstruction of data contained within it, or

within appropriate bound logbooks. This includes, but is not limited to: final data report pages, case narratives, chains of custody, raw data, digestion logs, any notes concerning client directives, observations of the samples, QC summary pages, and any other documentation required by verbal or written contract with the client. All software generated data are stored in hard copy format within the data report.

Hard copy data reports are retained for a period of no less than 5 years. Disposal of old records is carried out in such a way that any information traceable to a client or a specific sampling site is fully destroyed (e.g. shredded). Hard copy reports are stored on ventilated shelves in the laboratory storage closet, which has fire suppression devices available to it. All documents pertaining to data generation are stored in a safe and secure environment, and held in confidence to the client.

11.9 Electronic Data Control

Primary control of electronic data occurs at the physical security level, by preventing any non-authorized persons access to the premises without an escort. Secondary control of electronic data is achieved by employing only personnel with proven ethical understanding of data integrity. Tertiary data control at the instrument level is controlled by the software auditing mechanisms built into the major instrumental software utilized by the laboratory. Quaternary electronic data control is achieved by retaining hard copy records of all electronic data produced by the laboratory in appropriate data files.

11.10 Contract or Accreditation Specific Records

Contract or accreditation specific records shall be maintained for a period of time in keeping with the contractual or accreditation specific requirements. These documents are stored safely and securely, as all other documents are, and are available at all times to the accreditation authority or contract representative, usually a period of 5 years.

12.0 References

National Environmental Laboratory Accreditation Conference (NELAC, 2003). Program Policy and Structure

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- U.S. EPA 1992c. Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere. 40 CFR 50, Appendix J (770-774).
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- U.S. EPA. 1997. Reference Method for the Determination of Fine Particulate Matter as PM2.5 in the Atmosphere. 40 CFR 50, Appendix L. Federal Register, Vol. 62, No. 138, July 18, 1997 (57-95).
- U.S. EPA. 1998a. Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air. EPA 625/R-96/010a. U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, NC.
- U.S. EPA. 1998b. EPA Requirements for Quality Management Plans. EPA QA/R-2. U.S. Environmental Protection Agency, Quality Assurance Division, Washington, DC.
- U.S. EPA. 1998c. Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods. Quality Assurance Document 2.12. Human Exposure and Atmospheric Sciences Division, U.S. Environmental Protection Agency, Research Triangle Park, NC.

Appendix A

Staff Résumés

Paul D. Duda

President, Lead Project Manager, LIMS Administrator

Background:

Experience in air quality filter analysis by X-Ray Fluorescence; experience as project manager; experience with SAS, CLP, and CLP data package requirements. Experience as Laboratory Information Management System (LIMS) administrator, coordinating all LIMS activities; special expertise in interfacing laboratory data to client-specific databases and end-user data programs.

Career Chronology:

Employment Information

President, Lead Project Manager LIMS Administrator, Chester LabNet, Tigard, OR 2001 – present

Project Manager, LIMS Administrator, Sample Custodian, Chester LabNet, Tigard, OR 1992-2001.

Weighroom and X-Ray Fluorescence Technician, Chester LabNet, Tigard, OR 1989 - 1992.

1987 - 1988

Responsibilities and Duties

Corporate affairs for laboratory, including proposal writing, marketing and sales, program and project management, overall profit/loss for company, all accounting/payroll and purchasing; report production for all projects requiring EPA, CLP deliverables. Oversees all procedures, QA/QC, and corrective actions associated with sample receipt, log-in, chain-of-custody, and storage; project management and general and specialized report production; client management; oversees operation and maintenance of laboratory information management system (LIMS), including all software and hardware, general data entry, QA/QC, coordination with other project managers and technical staff, training of new users

Project management, all accounting/payroll and purchasing; report production for all projects requiring EPA, CLP deliverables. Oversees all procedures, QA/QC, and corrective actions associated with sample receipt, log-in, chain-of-custody, and storage; project management and general and specialized report production; client management; oversees operation of laboratory information management system (LIMS), including all software and hardware, general data entry, QA/QC, coordination with other project managers and technical staff, training of new users

Performed all operations of the filter gravimetry laboratory, including maintaining supplies, filter media acceptance testing, gravimetric analysis of filter media following EPA protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation. Also serves as XRF technician, including preparation of samples for analysis, instrument operation, interpretation of spectral results, QA/QC.

Miscellaneous employment.

Education:

Graduate Studies, Business Administration, Portland State University, Portland, OR, 1991-1992. B.S., Engineering Management, University of Portland, Portland, OR, 1987.

Sheri Heldstab

Inorganics Laboratory Manager & Technical Director, QA/QC Coordinator

Background:

Experience in inorganic environmental analytical chemistry; experience in method development of unusual sample matrices; experience in data interpretation and validation; experience with SAS and CLP data package requirements, experience in technical writing of internal technical documents; experience in NELAC QA compliance requirements.

Career Chronology:

Employment Information	Responsibilities and Duties	
Inorganic Laboratory Manager, QA/QC Coordinator Chester LabNet, Tigard, OR 1999 - present	Oversee all operations of the inorganics laboratory; ensure data meets QA/QC requirements; oversee and train other chemists; ensure meeting of due dates; analyze samples when sample load requires; and proper maintenance of instruments.	
	Oversight of standard operating procedure and program document system; QA review of data reporting; report production for all projects requiring EPA CLP deliverables, responsible for managing NELAC accreditation requirements.	
Account Manager, Lab Support, Portland, OR 1998 - 1999	Performed all duties required to run a one person branch office, including service calls, resolution of client disputes, marketing to new clients, filling of orders and recordkeeping.	
Chemist, ChemTrace, Portland, OR 1997 – 1998	Primary operator for IC and GFAA. Performed analysis on high purity water for various nutrients, microbiological testing and silica content.	
Lead Chemist, Chester LabNet, Tigard, OR 1994 – 1997	Primary operator for ICP, GFAA, CVAA. Analyzed variety of air quality samples using primarily CFR methods. Supervised Associate Chemist. Generated CLP QC reports. Managed sample throughput and Level I data validation of laboratory.	
Associate Chemist, Chester LabNet, Tigard, OR 1992 – 1994	Primary operator for IC. Analyzed variety of environmental samples using CFR, SW846, DW, SM, NIOSH, OSHA and a variety of other methods.	
Laboratory Technician, ASiMI, Washougal, WA 1991 – 1992	Analyzed high purity raw silicon for contaminants utilizing specialized equipment. Generated QC reports to be used in the preparation of Certificates of Lot Analysis.	
Chemist, Coffey Laboratories, Portland, OR 1990 - 1991	Analyzed a variety of environmental samples for inorganic constituents using DW, SW846, and SM methods.	

Education:

B.S., Biology (Chemistry minor), University of Oregon, 1989 Secondary Teaching Certification, University of Oregon School of Education, 1990

Lisa Ball

Project Manager, Sample Custodian

Background:

Experience as Project Manager. Experience as environmental analytical chemist.

Career Chronology:

Employment Information

Project Manager, Sample Custodian, Chester LabNet, Tigard, OR 2003 - present

Project Manager, Sample Custodian, Weighroom Coordinator, Chester LabNet, Tigard, OR 2001 - 2003

Chemist, Weighroom Coordinator, Chester LabNet, Tigard, OR 1997-2001

Extraction Chemist, Oregon Analytical Laboratory, Beaverton, OR, 1997 (full-time, temporary)

Chemist, American Environmental Network, Durham, OR, 1996-1997

Responsibilities and Duties

Project management. Performs all procedures, QA/QC, and corrective actions associated with sample receipt, log-in, chain-of-custody, and storage; project management and general and specialized report production; client management; monitors and performs daily and weekly LIMS backup; general data entry, QA/QC, coordination with other project managers and technical staff, training of new users.

Project management. Performed all procedures, QA/QC, and corrective actions associated with sample receipt, log-in, chain-of-custody, and storage; project management and general and specialized report production; client management; monitored and performs daily and weekly LIMS backup; general data entry. Oversee and perform all operations of the filter gravimetry laboratory.

Performed all operations of the filter gravimetry laboratory, including maintaining supplies, filter media acceptance testing, gravimetric analysis of filter media following EPA protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation. Analyzed air quality samples using primarily CFR methods, including: sample preparation and digestion and analysis of samples. Principal Operator of IC, ICP, GFAA, CVAA. Responsible for Level I data review and reporting.

Performed extractions for total petroleum hydrocarbons (TPH and TPHD), hydrocarbon identification (HCID), PAHs, and oil and grease. Extractions included separatory agitation, as well as distillations. Digested, extracted and analyzed water and soil samples for a variety of inorganic constituents including: CODs, pHs, alkalinity, open-cup flashpoints, Total Kjeldahl Nitrogen analysis. Cyanide distillation and analysis.

Primary wet chemist. Brought new wet chemistry methods on line and wrote corresponding SOPs for wet chemistry methods.

Education:

B.S., Integrated Science, Portland State University, 1996. OSHA 1910.120: 24-hour, 1996.

Anthony (Tony) Ochmanek

Lead Chemist, Weighroom Technician

Background:

Experience in environmental air quality analysis of air quality samples on a variety of matrices using CFR, SW846, NIOSH and OSHA methods for inorganic constituents. Experience in environmental analytical chemistry.

Career Chronology:

Employment Information

Lead Chemist, Weighroom Technician, Chester LabNet, Tigard, OR 2003 - present

Associate Chemist, Weighroom Technician, Chester LabNet, Tigard, OR 2001 - 2003

Quality Control Analyst,
Orasure Technologies
Beaverton, OR
(via Lab Support temporary agency)
2000 – 2001

Wet Chemistry Analyst Trace Analytical Laboratory Muskeegon, MI 2000

Responsibilities and Duties

Analyze variety of air quality samples using primarily CFR methods; perform sample preparation and digestion; primary operator for ICP, GFAA, IC, OC/EC; principal chemist for wet chemical analysis, performance of level I data review in real time; perform maintenance and repair of instrumentation; manage sample throughput, laboratory stocks and supplies.

Perform all operations of the filter gravimetry laboratory as described below.

Sample preparation and digestion, analysis of a variety of samples for ionic constituents by ion chromatograph (anions and cations), principal chemist for wet chemical analysis, metals digestions, performance of level I data review in real time, maintenance and repair of IC, principal OC/EC operator, back-up operation of the ICP, GFAA, and CVAA.

Perform all operations of the filter gravimetry laboratory, including filter media acceptance testing, gravimetric analysis of filter media following CFR protocols, all QA/QC and corrective actions, maintenance of log books and QC documentation.

Aided in development of Oral HIV testing device. Tested each component of device for functionality and longevity. Maintained all appropriate logbooks and documentation. Developed and prepared antibody and serum mixtures.

Digested, extracted and analyzed water and soil samples for a variety of inorganic and biological constituents including: CODs, BODs, TOCs, Phosphates, Hexavalent Chromium, TCLP extractions, Total Coliform, Total Kjeldahl Nitrogen analysis, Cyanide distillation and analysis. Maintained and repaired instruments. Maintained logbooks and spreadsheets. Responsible for Level I data review in real time. Completed internal reporting forms.

Education:

B.S. Biology, Marquette University, 1999

Richard H. Sarver

Principal XRF Scientist, XRF Technical Director

Background:

Experience in analytical chemistry, including biochemical applications and environmental air quality analysis, specializing in the analysis of air particulates by x-ray fluorescence.

Career Chronology:

Employment Information

Principal XRF Scientist, Chester LabNet, Tigard, OR 1986 - present.

Responsibilities and Duties

Coordinate all XRF activities with project managers as needed; train XRF technicians and oversee all XRF operations; market XRF capabilities to outside clients; responsible for maintenance and repair of instruments, supervise sample flow, data interpretation, QA/QC and report generation from XRF analysis; perform highly specialized sample preparation for non-deposit samples, including size fraction and resuspension; technical guidance for clients and in-house staff. Perform XRF analysis and provide technical assistance for state and federal agencies, industrial, consulting and university clients.

Awarded EPA equivalency method EQL-0589-072, "Determination of Lead Concentration in Ambient Particulate Matter by EDXRF Spectrometry in May 1989. Principal scientist for the XRF analysis of air particulates for the U.S. EPA national PM2.5 Chemical Speciation Program. Developed XRF method of analysis for the Hazardous Element Sampling Train (HEST), which used activated carbon to trap volatile metals. Continues to participate in the ongoing effort to obtain equivalent method status to EPA Method 29.

Analytical Chemist, Pioneer Hi-Bred International Portland, OR 1980-1986. Utilized FID/GC analysis to determine metabolic pathways of resident microorganisms in the digestive tract of stressed mice. Handled animals and performed analytical work. Developed SOPs for in house use.

Education:

A.A.S., Chemical Technology, Chemeketa Community College, Salem, OR 1980.

Selected Publications and Presentations:

Sarver, R. H. 1996. Aerosolization as a Means of Sample Preparation of Geological Materials for XRF Analysis and its Validity Compared to EPA Method 3050A Digestion. Journal of the Air & Waste Management Association. 46: 234-240.

Sarver, R.H. and Lytle, C.R. 2000. Parameter optimization for the analysis of PM2.5 by energy dispersive x-ray fluorescence (EDXRF). Presented at PM2000: Particulate Matter and Health, Air & Waste Management Association Specialty Conference, Charleston, SC, January 24-28, 2000.

Sarver, R.H., Mace, J.C. and Duda, P.D. 2002. XRF: Inter-Excitation Quality Assurance and Deposit Uniformity. Presented at Symposium on Air Quality Measurement Methods & Technology, Air & Waste Management Association Conference, San Francisco, CA, November 13–15, 2002.

Appendix B

Listing of Chester LabNet Standard Operating Procedures (SOPs)

SOP#	SOP Title
AD-001.03	General Administrative Procedures
AD-002.02	Waste and Sample Disposal
AD-003.02	Refrigerated Storage Monitoring
AD-004.02	Glassware Cleaning for Inorganics Laboratory
AD-005.03	Reagent Procurement and Control
AD-006.03	Laboratory Deionized Water Supply
AD-007.03	Laboratory Information Management System (LIMS)
AD-008.04	Sample Receipt and Log-In
GR-001.04	8x10 Quartz & Glass Fiber Filter Inspection and Gravimetry
GR-001a.02	Punching of Exposed 8x10" Quartz or Glass Fiber Filters
GR-002.04	80-125 mm Filter Inspection and Gravimetry
GR-003.02	Gravimetric Processing of 25-47mm Quartz Filters **DEACTIVATED 2/02**
GR-004.03	Chemical Impregnation of Cellulose Filters
GR-005.02	Impregnation of Cellulose Filters with Sodium Carbonate **DEACTIVATED 1/02**
GR-006.04	Filter Cassette Loading and Unloading
GR-007.02	Inspection & Preparation of 25-47mm Teflon Filters **MERGED W/GR-010, 5/03**
GR-008.02	Oil Coating of Teflon Filters
GR-009.01	Inspection & Preparation of 82.6-125mmFilters **DEACTIVATED 3/02**
GR-010.03	Teflon & Quartz Fiber Filter Preparation and Gravimetry (25mm, 37mm & 47mm)
	Inspection and Preparation of Carbon Impregnated Filters **SUSPENDED
GR-011.02	6/05**
GR-012.01	Inspection & Preparation of 102mm Teflon Filters **DEACTIVATED 3/02**
GR-013.02	Inspection & Preparation of 8x10" Pallflex Weave Filters **DEACTIVATED 1/02**
GR-014.01	Gross Weighing of 25-47mm Teflon Filters **DEACTIVATED 3/02**
GR-015.01	Quartz Filter Preparation for Carbon Analysis
GR-016.02	Preparation & Use of Control Charts for Gravimetric Analysis
GR-017.01	Acceptance Testing of 47mm Teflon Filters (Drop Test)
IC-001.02	Borate Eluant Anions ** DEACTIVATED 4/00 **
IC-002.02	Preparation of Air Filters for Fluoride Analysis ** DEACTIVATED 4/00 **
IC-003.03	Extraction of Filter Media for Ion Chromatographic Analysis
IC-004.02	Clean-Up of Anion Columns **DEACTIVATED 1/02**
IC-005.04	Ion Chromatography: Anions
IC-006.04	Ion Chromatography: Cations
IC-007.02	Clean-Up of Cation Columns **DEACTIVATED 1/02**
ME-001.03	Analysis of Elements by ICP-AES (P40) **DEACTIVATED 3/03**
ME-002.04	Analysis of Elements by Graphite Furnace Atomic Absorption (Aanalyst 600)
ME-003.04	Sample Digestion for Analysis of Elements by ICP or GFAA
ME-004.02	Analysis of Mercury in Aqueous Samples
ME-005.02	Analysis of Mercury in Solid Samples
ME-006.03	Analysis of Mercury in Hopcalite Sorbent Tubes
ME-007.01	Analysis of Elements by Inductively-Coupled Plasma Emission (Optima 2000)
00.651.55	
OC-001.02	Organic & Elemental Carbon by the Thermal-Optical Method

SOP#	SOP Title
XR-001.01	Resuspension of Particulate Matter onto Filter Media
XR-002.02	Analysis of Elements in Air Particulates by X-Ray Fluorescence (Kevex 770 & 772)
XR-003.01	Preparation of Samples for Resuspension
XR-004.01	Kevex XRF Spectrometer Calibration
XR-005.01	Kevex Spectrometer Data Generation, Interpretation and Reporting
XR-006.01	Analysis of Elements in Air Particulates by X-Ray Fluorescence (Kevex-771)
QA-001.04	Laboratory Training
QA-002.03	Laboratory Data and Report Validation
QA-003.04	Implementation, Distribution, & Control of Std. Operating Procedures
QA-004.03	Distribution and Control of Laboratory Notebooks
QA-005.02	Control of Laboratory QA/QC Records **DEACTIVATED 9/11/01**
QA-006.03	Determination of Detection Limits, Precision & Bias & DoC
QA-007.02	Calibration of Laboratory Pipettes
QA-008.02	Assembly and Preparation of Data Reports (Original QA-008 merged with QA-002)
ST-001.01	Halide & Hydrogen Halide Emissions from Stationary Sources
ST-001.01	Particulate Emissions from Stationary Sources
ST-002.01 ST-003.02	Sulfur Dioxide Emissions from Stationary Sources
ST-003.02 ST-004.02	, , , , , , , , , , , , , , , , , , ,
ST-004.02 ST-005.02	Nitrogen Oxide (NOx) Emissions from Stationary Sources **SUSPENDED 4/05** Sulfuric Acid Mist & Sulfur Dioxide Emissions from Stationary Sources
ST-005.02 ST-006.02	Elements by EPA Method 29 or CARB Method 436
ST-000.02 ST-007.01	Hydrogen Sulfide Content of Fuel Gas Streams
ST-007.01 ST-008.01	
ST-008.01 ST-009.01	Inorganic Lead from Stationary Sources Total Fluoride Emissions - Specific Ion Selective Electrode Method
ST-009.01 ST-010.03	Total Reduced Sulfur Emissions Total Reduced Sulfur Emissions
ST-010.03	Total Reduced Sulfur ** DEACTIVATED** 6/05 (merged w/ ST010 6/14/05)
ST-011.02 ST-012.01	Sulfur Dioxide Emissions **DEACTIVATED ** 8/05
ST-012.01	Particulate & Gaseous Mercury
ST-013.02 ST-014.01	Beryllium Screening
ST-015.01	Beryllium Emissions from Stationary Sources
ST-016.01	Mercury in Sewage Sludge **SUSPENDED** 6/05
ST-010.01	Particulate & Gaseous Arsenic Emissions **SUSPENDED** 6/05
ST-017.01	Ammonia in Stationary Sources
	,
WC-001.01	Chemical Oxygen Demand (COD) **SUSPENDED** 1/05
WC-002.01	Specific Conductance **SUSPENDED 1/05**
WC-003.01	Fluoride by Ion Selective Electrode
WC-004.01	Ammonia-Nitrogen by Ion Selective Electrode **DEACTIVATED** 10/05
WC-005.01	Gravimetric Oil & Grease in Liquids ** DEACTIVATED ** 4/05
WC-006.01	Soil Ph **SUSPENDED** 1/05
WC-007.01	Gravimetric TPH. ** DEACTIVATED ** 4/00
WC-008.01	Alkalinity. **SUSPENDED** 1/05
WC-009.01	Cation Exchange Capacity. **SUSPENDED** 1/05
WC-010.01	Redox Potential (eH). **SUSPENDED** 1/05
WC-011.01	Hardness. **SUSPENDED** 1/05
WC-012.01	Nitrite-Nitrogen. **SUSPENDED** 1/05
WC-013.01	Organic Matter. Walkley-Black Method **SUSPENDED** 1/05
<u>SOP #</u>	SOP Title

WC-014.01	pH. **SUSPENDED** 1/05
WC-015.01	Phosphorous/Phosphate, All Species **SUSPENDED** 1/05
WC-016.01	Total Dissolved Solids. **SUSPENDED** 1/05
WC-017.01	Total Kjeldahl Nitrogen. **DEACTIVATED** 10/05
WC-018.01	Total Suspended Solids. **SUSPENDED** 1/05
WC-019.01	Turbidity. **SUSPENDED** 1/05
WC-020.01	Hexavalent Chromium. **SUSPENDED** 1/05

Appendix C

Listing of Chester LabNet Utilized Methods (as listed by issuing authority and method number)

NELAC Accredited Method(s)

Method Number	Analyte/Element	<u>Instrumentation</u>
CARB SOP MDL 039	Hexavalent Chromium	IC-PCD

US EPA IO methods

Method Number	Analyte/Element	<u>Instrumentation</u>
2.1	TSP & PM ₁₀	gravimetric
2.2	PM ₁₀	gravimetric
2.3	PM ₁₀	gravimetric
3.1	metals prep.	wet chemical
3.2	metals	GFAA
3.3	metals	XRF
3.4	metals	ICP

40 CFR 60 Source Testing methods

Method Number	Analyte/Element	<u>Instrumentation</u>
5	Particulates	balance (gravimetry)
6	SO ₂	titrimetric
7	NO _x	IC
8	H ₂ SO ₄ /SO ₂	titrimetric
11	H ₂ S	titrimetric
12	Pb	ICP
13	F	IC
14	F	IC
15a	Reduced Sulfur	titrimetric
16a	Reduced Sulfur	titrimetric
20	SO ₂	titrimetric
26	H _X , H _X & X ₂	IC
29	Metals	ICP
101	Hg	CVAA
102	Hg	CVAA
103	Be	ICP
104	Be	ICP
105	Hg	CVAA
108	As	ICP
201/202	Particulates	balance (gravimetry)
306	Cr	ICP or GFAA
CTM 027	NH ₃	IC

NIOSH methods

Method Number	Analyte/Element	<u>Instrumentation</u>
0500	Particulates	balance (gravimetry)
0600	Particulates	balance (gravimetry)
6001	Arsine	GFAA
6004	SO ₂	IC
6006	Diborane	ICP
6009	Hg	CVAA
6011	Br ₂ & Cl ₂	IC
6014	NO_2	IC
6016	NH ₃	IC
7300	metals	ICP
7600	Hexavalent Cr (CrVI)	wet chemical
7902	F	IC
7903	Inorganic Acids	IC

CARB methods

Method Number	Analyte/Element	<u>Instrumentation</u>
005	As	GFAA
421	HF & HCI	IC
423	As	GFAA
425	Total Cr	GFAA
425	Hexavalent Cr (CrVI)	wet chemical
436	multiple metals	ICP
SOP MLD039	Hexavalent Cr (CrVI	IC

EPA SW-846 methods

Method Number	Analyte/Element	<u>Instrumentation</u>
3050	metals prep.	wet chemical
6010	metals	ICP
7000 series	metals	GFAA
7470/7471	Hg	CVAA
9045	рН	pH meter
9050	conductivity	conductivity probe
9080/9081	cation exchange capacity	ICP/wet chemical

ODEQ methods

Method Number	Analyte/Element	<u>Instrumentation</u>
5	Particulates	gravimetric
8	Particulates	gravimetric

EPA Water/Wastewater methods

Method Number	Analyte/Element	<u>Instrumentation</u>	
120.1	Conductance	conductivity probe	
130.2	Hardness	titrimetric	
150.1	рН	pH electrode	
160.x	solids	gravimetric	
180.1	Turbidity	Nephlometric	
200.0	metals prep.	wet chemical	
300.0	Anions	IC	
300.7	Cations	IC	
305.1	Acidity	titrimetric	
310.1	Alkalinity	titrimetric	
340.2	F	Ion Selective Electrode	
350.3	NH ₄	Ion Selective Electrode	
351.4	TKN	Ion Selective Electrode	
354.1	NO ₂	colorimetric	
365.3	PO ₄ forms	colorimetric	
376.1	Sulfide (H ₂ S)	titrimetric	
377.1	Sulfite (SO ₃)	titrimetric	
410	COD	colorimetric	

Appendix D

Data Integrity and Personal Ethics Policy (including personnel agreement page)

Appendix D: Personal Ethics and Data Integrity Policy

D.1 Introduction

Our goal is to provide the most informed and accurate inorganic analysis of air quality samples available from a commercial laboratory. Chester LabNet's management is committed to good professional practice and to the quality of its environmental testing in servicing its clients. To achieve this goal, it is critical that all employees understand the need for honesty and full disclosure of variances in all arenas of analyses performed, when and how to report data integrity issues, and the documentation of such issues when they arise.

Data integrity is defined as data of known quality, analyzed by documented procedures, fulfilling all Quality Control standards established with those procedures, and meeting the requirements of the client. Inherent in the concept of data integrity is that no false manipulations of data or samples, or omissions of pertinent information, be performed to meet the Quality Control criteria. This inherent need is governed by the personal ethics of each employee and the overall corporate culture of Chester LabNet.

D.2 Management Responsibilities

Management responsibilities are many, and begin with creating a culture of trust and honesty within the organization. Technical directors of each department understand that employees are human and do make mistakes. Honest mistakes are corrected and addressed to the employee. Technical Directors are charged with upholding the intent of this policy and implementing the specific requirements not only of this policy, but also of each documented procedure practiced by the laboratory. In addition, Technical Directors must perform their oversight duties with a positive attitude, maintaining focus on the goal of producing high quality data, and without personal attacks or negative attitudes which might lower morale or decrease the likelihood of employees being open and honest. Managers and Technical Directors must do their utmost to encourage a corporate culture of honesty and security for each employee, such that no employee is ever afraid to bring forth issues or problems they might encounter.

Technical Directors monitor the adherence of this document by supervising their employees and the data and/or reports produced by their employees. Evidence of unethical behavior such as improper manipulations of data, clock rolling, inappropriate changes in concentrations of standards, failure to follow written procedures to bypass Quality Control checks, insufficient documentation, etc, are addressed to the employee and are documented via the annual review or addendums to the annual review, which are kept in the employee's personnel file. Technical Directors are charged with monitoring the breach after

such a discussion to ensure that the employee's behavior has changed. If no change has occurred, the Technical Director and President shall decide upon what is considered appropriate action to be taken. Actions may include termination of the employee, moving the employee to a different department, revoking some of the employee's duties or other actions to resolve the issue and prevent its further occurrence. The worst-case scenario may result in criminal or civil prosecution of the individual employee, fines or possible jail sentences. The laboratory does not and will not defend any employee charged in a court of law who, despite management's best efforts, knowingly submits false, incomplete or flawed data.

D.3 Employee Responsibilities

"Employees" include both managerial and non-managerial staff. Employee responsibilities include following written procedures and known scientific principles to produce data of the highest degree of scientific defensibility possible within the limitations of the sample matrices and currently available instrumentation. Each employee is responsible for ensuring that the data and/or reports they produce are accurate and complete, and meet the Quality Control criteria described in the method or written procedure for the task. Employees must also read section 3.3.2.2 of this document.

An employee's personal ethics play a large role in maintaining data integrity. While ethics are more difficult to define, and certainly more difficult to instill and enforce, for the purposes of this document, the most fundamental ethic required by Chester LabNet is honesty. Lying, either by data manipulation, verbal falsification of procedures followed, or by omission, is not supported in any way by Chester LabNet.

All employees are charged with reporting any data integrity issue, be it their own or that of others to their technical director or supervisor in a timely manner. If that person seems unresponsive, employees should report their concern to either another related technical director or to the company president. Non-reporting of known breaches of ethics is considered equally as damaging as having performed the breach oneself, and is subject to the same consequences as described in section D.2.

Breaches of ethical behavior include, but are not limited to:

- Blatant falsification of data
- Improper data manipulations, such as questionable hand integrations, peak shaving, undocumented blank subtractions, not following established rounding rules in order to meet quality control criteria, etc
- Changing computer clocks to show a different time in order to meet holding time criteria

- Changing standard or QC sample concentrations to force them to meet QC criteria (e.g. diluting or spiking LCSs)
- Failure to record information as described in the relevant SOPs (e.g. not recording balance calibration data or temperature/humidity during gravimetric analysis, etc.)

Given the wide variety of matrices, sampling methods, background contaminants and physical states of samples analyzed at Chester LabNet, it is to be expected that Quality Control criteria will occasionally fail. It is the responsibility of the employee to properly document the failure, attempt to meet the Quality Control criteria where possible, and ensure the client is informed of such deviations from normal protocol.

Any errors must be lined out with a single line, such that the original entry is still legible. The line out must be dated and initialed by the employee correcting the mistake. If the mistake is not obvious (such as a typographical error on a sample ID, or an incorrect date), the analyst must note why the correction or deletion has been performed.

In cases where non-conforming data is submitted to a client anyway, the client must be notified, usually in the case narrative, as to the nature of the non-conformance and the reason(s) and/or opinions as to why the non-conformances could not be rectified.

Other observations of samples, such as possible interfering peaks, mass changes as a result of filter defects, precipitation occurring during sample preparation which are out of the norm for a given method or any other observance which is not typical for a particular method must be noted in the raw data and the case narrative. Any opinions of the laboratory concerning data quality, integrity, accuracy or legal defensibility must be clearly documented, and must be noted to be the opinion of the laboratory. This documentation must be contained in the case narrative or conveyed to the client by some written means.

D.4 Summary

Chester LabNet endeavors to foster an open and non-retaliative corporate atmosphere where all employees are not only encouraged, but also expected, to bring any data integrity issues to the notice of the appropriate personnel. All employees understand the need to produce the highest quality data possible. While management holds the ultimate responsibility for data integrity issues, it is the personal ethics of every employee that supports the production of high quality data, and thereby the reputation of the company as a laboratory interested in providing the best data possible to their clients.

Personal Ethics and Data Integrity Agreement Page

The following approved signatories, by their signature, attest to having read, understood and agreed to the most current version of the Personal Ethics and Data Integrity Policy for Chester LabNet:

<u>Name</u>	Title/Responsibility	<u>Signature</u>	<u>Date</u>
Paul Duda	President, Chester LabNet, LIMS/RTRAC Administrator Customer Service Technical Director		
Sheri Heldstab	QA/QC Coordinator Inorganics Lab Manager Inorganics Lab Technical Director		
Rick Sarver	XRF Technical Director Senior XRF Scientist		
Lisa Ball	Project Manager, Sample Custodian Weighroom Coordinator		
Tony Ochmanek	Lead Chemist Weighroom technician		



APPENDIX B

SOIL, SOIL VAPOR, AND CONCRETE SAMPLING AND ANALYSIS PLAN

Soil, Soil Vapor, and Concrete Sampling and Analysis Plan

Former Pechiney Cast Plate, Inc., Facility 3200 Fruitland Avenue, Vernon, California

Prepared for:

Pechiney Cast Plate, Inc.

Prepared by:

Geomatrix Consultants, Inc.

510 Superior Avenue, Suite 200 Newport Beach, California 92663 (949) 642-0245

July 20, 2007

Project No. 10627.003.0





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FIGURE

Figure 1 Excavation Sample Grids with Sample Location Layout



SOIL, SOIL VAPOR, AND CONCRETE SAMPLING AND ANALYSIS PLAN

Former Pechiney Cast Plate, Inc., Facility 3200 Fruitland Avenue Vernon, California

1.0 INTRODUCTION

Sampling and analysis procedures are described below for confirmation soil and concrete sampling. If additional characterization work is necessary, additional soil, soil vapor and concrete sampling will be completed as described is this Plan. This Plan will be used in conjunction with the Feasibility Study/Remedial action Plan (FS/RAP) (Geomatrix, 2007a), Below Grade Demolition Plan (Geomatrix, 2006a), below-grade technical specifications, and other related documents related to this project.

2.0 CONFIRMATION AND CHARACTERIZATION SAMPLING

Confirmation and characterization soil, soil vapor, and concrete sampling will be conducted by a Geomatrix Consultants, Inc. (Geomatrix), field geologist/engineer under the supervision of a Professional Geologist or Professional Engineer. Site health and safety planning, utility clearance, sampling and analysis, sample handling procedures, equipment decontamination and waste contaminant procedures, and sample location recording are described herein.

2.1 Site Health and Safety Plan and Utility Clearance

A Site-specific Health and Safety Plan (HASP) will be used during the field activities performed by Geomatrix personnel (Geomatrix, 2006b). The HASP will address the potential risks to the personnel performing the sampling activities proposed in this Plan.

Utility locating and clearance will be the responsibility of the demolition contactor managing the Site.

2.2 Soil Sampling and Analysis

Soil samples will be collected for confirmation and characterization purposes from areas where impacted soil is removed and/or observed during the below-grade demolition and remediation activities. The soil samples will be collected directly from the backhoe bucket of the excavating equipment. In some cases, soil samples may also be collected using hand auger or other drilling methods.



The number of confirmation soil samples collected and analysis selected will be determined by Geomatrix. The analytical suite will be selected based on field observations, a review of past operations in the area, and results of previous investigations in the vicinity of impacted soil. These samples will be analyzed under 24- to 48-hour turnaround to support the demolition activities. The suite of analyses may include one or more of the following test methods:

- Total Petroleum Hydrocarbons (TPH) with carbon chain range quantification using Environmental Protection Agency (EPA) Method 8015M (Modified);
- Volatile Organic Compounds (VOCs) using EPA Method 8260B and field preservation Method 5035;
- Polychlorinated Biphenyls (PCBs) using EPA Method 8082;
- California Assessment Manual Metals using EPA Methods 6010B/7000; or
- Semi-Volatile Organic Compounds (SVOCs) using EPA Method 8270C. Samples will be selected for SVOC analysis based on the reported TPH concentrations; soil samples exhibiting greater than 2,000 mg/kg of TPH will be analyzed for SVOCs.

Additional characterization soil samples may be collected based on observations made during demolition and soil removal and will be included as part of the field quality assurance/quality control (QA/QC) program for the Project. The QA/QC procedures are discussed in the Quality Assurance Project Plan (QAPP; Geomatrix, 2007b).

Waste profile sampling of investigative derived waste (IDW) will also be conducted. Sampling and handling procedures will be determined when the waste is generated.

2.3 Soil Confirmation Sample Locations

In general, confirmation samples will be collected from "small area" excavations (less than 100 cubic yards of soil) by dividing the excavation into four equal parts using a grid pattern. A typical grid pattern for a small excavation is shown on Figure 1. Four side wall soil samples will be collected, one on each sidewall at the location where the gird line intersects the sidewall (horizontal locations). At the grid line point on the wall, the sample location will be placed at a vertical midpoint between the top and bottom of the excavation wall. At the base of the excavation, two soil samples will be collected from areas located in diagonally opposite grids of the four grid squares, equally representing the excavation bottom.

In general, confirmation samples will be collected from "large area" excavations (greater than 100 cubic yards of soil) by dividing the excavation into at least six equal parts in a grid pattern.



A typical grid pattern for a large excavation is shown on Figure 1. At least six side wall soil samples will be collected along the side walls at the location where the gird line intersects the wall (horizontal). At the grid line point on the wall, the sample location will be placed at a vertical midpoint between the top and bottom of the excavation. At least two sidewall samples will be collect from the longer walls and at least one side wall sample will be collect from the shorter wall. At a minimum, confirmation samples will be spaced horizontally at a distance of at least 10 to 15 feet along the side walls. At the base of the excavation at least three soil samples will be collected from areas located in diagonally opposite grids of the six grid squares, equally representing the excavation bottom. The actual number of confirmation samples collected from the "large area" excavation will be determined in the field based on the size of the excavation.

2.4 Soil Vapor Sampling and Analysis

A soil vapor sampling will be conducted in portions of the Site, including within the area of Building 112 (Stoddard solvent impacted soil). The sampling will be used to assess the concentration and distribution of vapor-phase VOCs (if present at the site) and Stoddard solvent. Temporary soil vapor points will be installed using hydraulic-drive, direct-push installation methods. Vapor samples will be collected at each location from approximate depths of 5 and 15 feet below ground surface.

The soil vapor sampling will be conducted in general accordance with California Regional Water Quality Control Boards, Los Angeles Region (RWQCB) "Interim Guidance for Active Soil Gas Investigation" dated on February 25, 1997 and the Department of Toxic Substances Control (DTSC) and RWQCB "Advisory-Active Soil Gas Investigations" dated January 28, 2003 (Joint Advisory).

Soil vapor samples will be analyzed by an on-site mobile laboratory for the RWQCB target list of 23 VOCs, 2-butanone, naphthalene, and Stoddard solvent using gas chromatography/mass spectrometry methods similar to EPA Test Method 8260B for soil and groundwater. For the mobile laboratory to report Stoddard solvent, the laboratory instrument will be calibrated against a Stoddard solvent calibration standard.



Target List of Cor	npounds for Soil Vapor Survey
1,1,1,2-Tetrachloroethane	Ethylbenzene
1,1,1-Trichloroethane (1,1,1-TCA)	Freon® 11 (Trichlorofluoromethane)
1,1,2,2-Tetrachloroethane	Freon® 113 (1,1,2-trichloro-1,2,2-trifluoroethane)
1,1,2-Trichloroethane (1,1,2-TCA)	Freon® 12 (Dichlorodifluoromethane)
1,1-Dichloroethane (1,1-DCA)	Methylene chloride
1,1-Dichloroethene (1,1-DCE)	Xylene (o, m, and p)
1,2-Dichloroethane (1,2-DCA)	Tetrachloroethene (PCE)
Benzene	Toluene
Carbon tetrachloride	trans-1,2-Dichloroethene (t-1,2-DCE)
Chloroethane	Trichloroethene (TCE)
Chloroform	Vinyl chloride
cis-1,2-Dichloroethene (c-1,2-DCE)	

In addition, the soil vapor sampling will include the following additional procedures.

- Hydrated bentonite will be used to achieve a seal at the surface of the temporary sampling probe. At each sample point, isopropyl alcohol will be used as an ambient air leak detection compound.
- A one time purge volume test will be conducted at the beginning of the work.
- Prior to sample collection, each sample point will be purged using the volume selected during the purge volume test, as per RWQCB guidelines. If no-flow or low-flow conditions occur, the soil vapor sample probe will be pushed deeper and another sampling attempt will be made.
- Ambient air blanks will be collected and analyzed during each day of sampling.
- Duplicate soil vapor samples will be collected at a frequency of 10 percent of the total samples collected.

Reporting limits for target soil vapor compounds will be consistent with the RWQCB reporting limits of 0.1 to 1 μ g/L. However, higher reporting limits may result if compounds have concentrations greater than the calibration range and require dilution.

2.5 Concrete Characterization Sampling

Concrete characterization testing has been previously conducted at the Site and the data are summarized Appendix A of the FS/RAP. Based on these data, PCB-impacted concrete slab areas where concentrations exceed site-specific remediation goal for PCBs will be demarcated



in the field by marking the slab surface. Impacted concrete will then be saw cut, removed, and transported off-site for disposal at an appropriate landfill facility.

Additional concrete characterization sampling will be conducted during the below grade slab removal work if visual stained concrete is observed beyond areas already tested for PCBs. Samples of the concrete will be collected from a core measuring 1.2-inches in diameter to a maximum depth of 3 inches into the concrete slab. The cores will be sent to the laboratory for crushing and will be analyzed for PCBs using EPA Method 8082.

Waste profile sampling of IDW will also be conducted needed at the time the materials are generated.

3.0 SAMPLE HANDLING PROCEDURES

Sample handling procedures applicable to this work will include sample containers and preservation, sample labeling, sample packaging, shipment, and chain-of-custody procedures; they are described in the following subsections.

3.1 Sample Containers and Preservation

Soil samples will be collected in acetate liners, 6-inch long brass or stainless steel sleeves, glass jars (glass jars will be not be used for samples being analyzed for VOCs or SVOCs), or volatile organic analysis vials. No preservatives are required for soil samples collected and submitted for TPH, SVOCs, metals, or PCBs (including concrete) analyses. Soil samples for VOC analysis will be collected using cut syringes (or equivalent) as described in EPA Preservation Method 5035 (field preservation). Soil samples for VOC analysis will be collected in cut syringes following EPA Method 5035 for field sample preservation. Soil samples will be preserved in the field using pre-weighed laboratory sample containers with methanol and sodium bisulfate preservatives. If the soil samples react with the sodium bisulfate preservative, it will be replaced with laboratory grade water. During the Phase II Environmental Site Assessment (ESA), soil from the Site reacted with the sodium bisulfate preservative, and it was replaced with laboratory grade water.

Clean, pre-packaged glass jars and containers will be provided by the laboratory. Samples, once packaged and labeled, will be placed in an ice-filled chest for transport to the stationary laboratory.



3.2 Sample Labeling

Sample identification will include a sample-specific identification code linking the sample to descriptive information recorded in field documents. A separate label will be affixed to each sample container with a self-adhesive backing. The sample identification code will consist of the following components:

- sequential sample location number (1, 2, 3, etc.);
- two letter code, describing the type of sample (SS = soil sample; SV = soil vapor sample; SM = sediment or sludge sample; DC = concrete sample; and DW = decontamination water sample); and
- two-digit sequential number describing the sampling depth (01 = the first sample colleted at 5 feet below grade, 02 = the second sample collected at 10 feet below grade, etc.) or sequential sample from a side wall of an excavation (01 = the first side wall sample collected from the east wall, etc.).

As an example, a sample labeled 01-SS-01 would represent a soil sample collected from side wall sample location number 1, at about 6 inches into the side wall. Sample labeled 02-SS-01 would represent a soil sample collected from the bottom of the excavation at sample location 2, at a depth of 6-inches below the base of the excavation.

3.3 Sample Packaging, Shipment, and Chain-of-Custody

Soil and field QA/QC samples will be collected and will remain sealed within the sampling containers until analysis is conducted by the laboratory. Ice contained in resealable plastic bags will be placed in the ice chest and used to keep the samples chilled. The condition of samples will be inspected prior to shipment.

Chain-of-custody (COC) procedures will be followed to ensure field sample integrity and tracking of sample custody. Each time a sample changes hands, both the sender and the receiver will sign and date the COC form. When a sample shipment is sent to the laboratory, the top signature copy is enclosed in plastic and secured to the inside of the sample shipment containers. A COC record will be completed for each shipping container.

4.0 EQUIPMENT DECONTAMINATION AND WASTE CONTAINMENT

Sampling equipment (hand augers, shovels, etc.) will be re-used between sample locations. To reduce the potential for cross-contamination, re-usable sampling equipment will be decontaminated using the following procedure:



- 1. wash and scrub in the non-phosphate detergent and potable water (first bucket);
- 2. rinse or soak in potable water (second bucket);
- 3. rinse in DI water (third bucket); and
- 4. final rinse with DI water and air dry.

Disposable nitrile gloves will be worn during all decontamination activities. Decontamination water will be temporarily stored in 5-gallon buckets and transferred to a 55-gallon labeled drum at the end of each day. Decontamination of the backhoe bucket will be based on visual observation and the condition of the soil excavated prior to sampling.

Decontamination water temporarily will be stored on-site in Department of Transportation - approved 55-gallon labeled drums until the IDW have been characterized for waste management or disposal.

5.0 EXCAVATION AND SAMPLE LOCATIONS

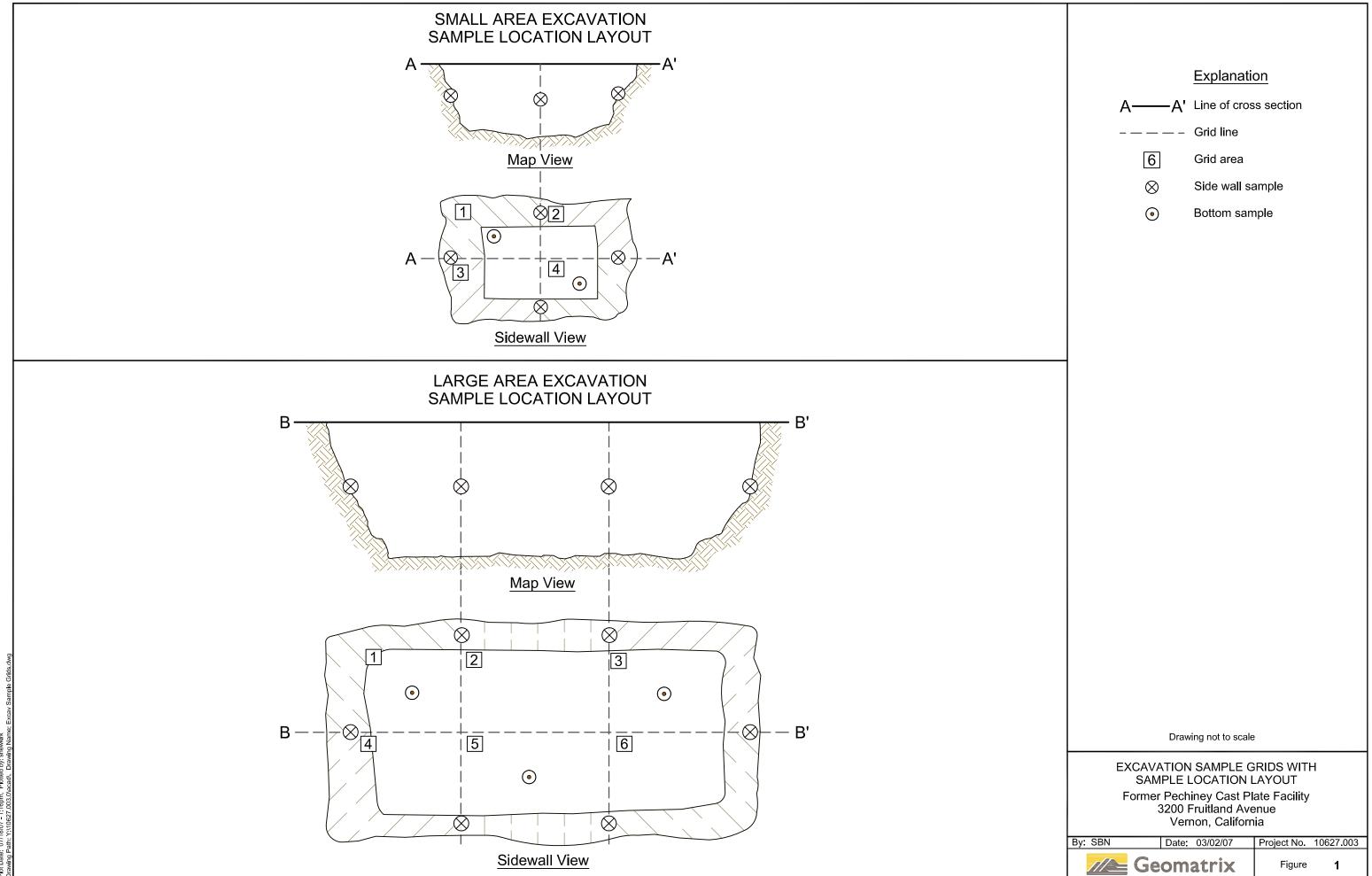
At the completion of the confirmation and characterization sampling and prior to excavation backfill, the perimeter of the excavation and sample points (when accessible) will be surveyed (vertical and horizontal control) by a licensed surveyor. If the sample points are not accessible to the surveyor, the confirmation soil samples will be measured in the field with respect to a corner of the excavation.

6.0 REFERENCES

- Department of Toxic Substances Control (DTSC), 2003, Advisory-Active Soil Gas Investigations (Advisory), January 13.
- Geomatrix Consultants, Inc., 2006a, Below Grade Demolition Plan, Former Pechiney Cast Plate Facility, Vernon, California, December.
- Geomatrix Consultants, Inc., 2006b, Site Health and Safety Plan, Pechiney Cast Plate Facility, Vernon Facility, 3200 Fruitland Avenue, Vernon, California, July.
- Geomatrix Consultants, Inc., 2007a, Feasibility Study/Remedial Action Plan, Former Pechiney Cast Plate Facility, Vernon, California, July.
- Geomatrix Consultants, Inc., 2007b, Quality Assurance Project Plan, Former Pechiney Cast Plate Facility, Vernon, California, July.
- Regional Water Quality Control Board, 1997, Interim Guidance for Active Soil Gas Investigation, reissued February 25.



FIGURES





APPENDIX C

PERIMETER AIR SAMPLING PLAN

Perimeter Air Sampling Plan Demolition and Remediation Activities

Former Pechiney Cast Plate, Inc., Facility 3200 Fruitland Avenue, Vernon, California

Prepared for:

Pechiney Cast Plate, Inc.

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July 2006 (Revised July 20, 2007)

Project No. 10627.003.0





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PERIMETER AIR SAMPLING PLAN FOR DEMOLITION AND REMEDIATION ACTIVITIES

Former Pechiney Cast Plate, Inc. Facility 3200 Fruitland Avenue, Vernon, California

1.0 INTRODUCTION

On behalf of Pechiney Cast Plate, Inc., Geomatrix Consultants, Inc. (Geomatrix), has prepared this Perimeter Air Sampling Plan (Plan) to be implemented during demolition and remediation activities at the former Pechiney Cast Plate, Inc. Facility in Vernon, California. The objective of this Plan is to collect data at the perimeter of demolition and remedial activities that will provide information on PM-10 particulate; lead; polychlorinated biphenyls (PCBs); trichloroethene (TCE); tetrachloroethene (PCE); benzene; 1,2,4-trimethylbenzene; and 1,3,5-trimethylbenzene emissions. Based on samples of soil and building materials, these are the key chemicals of concern that may be present in emissions during demolition and/or remedial activities. This Plan was updated in January 2007 to address below grade demolition and remediation activities and potential chemicals of concern associated with these activities. This Plan will be used in conjunction with the Feasibility Study/Remedial Action Plan (FS/RAP) (Geomatrix, 2007a), Below Grade Demolition Plan (Geomatrix, 2006a), below-grade technical specification, and other related documents for this project.

This Plan does not cover air monitoring for on-Site workers. Worker exposure monitoring is the responsibility of the demolition contractors performing the structural demolition and remediation work. Monitoring during asbestos abatement is being addressed by the abatement contractor and abatement monitor (Aurora). A Site Health and Safety Plan has been prepared for monitoring potential exposure by Geomatrix employees (Geomatrix, 2006b).

2.0 SITE BACKGROUND

The Vernon facility is located at 3200 Fruitland Avenue on an approximately 26.9 acre parcel (Figure 1). The facility consists of office and manufacturing buildings occupying approximately 600,000 square feet of the Site. The remaining areas are parking lots, outside storage areas, and partially paved vacant lots. The Vernon facility is surrounded by a fence with the Site entrance located on Fruitland Avenue.



2.1 SITE HISTORY

Aluminum Company of America's (Alcoa) operations at the Pechiney Cast Plate Facility reportedly began in approximately 1937. In approximately 1997, Alcoa sold the eastern half of its facility, which subsequently was razed, subdivided, and redeveloped as industrial and commercial properties. In December 1998, Alcoa sold the western portion of the facility (3200 Fruitland Avenue) to Century Aluminum Company. In 1999, Pechiney Cast Plate, Inc., purchased the Site.

The Vernon facility was used to manufacture high-precision cast aluminum plates and lies within an area zoned for industrial and commercial use. The facility is no longer in operation.

2.2 CHEMICALS OF POTENTIAL CONCERN

PCBs, metals (specifically lead as well as other metals), and volatile organic compounds (VOCs) have been detected in soil at the Site. TCE and PCE were the primary VOC detected in soil vapor and soil at the Site above screening levels for human health and/or potential impact to groundwater within buildings 106, 108 and 112. Metals, with the exception of arsenic, detected in soil did not exceed screening levels and/or background; however, soluble concentrations of some metals exceeded hazardous waste criteria. PCB concentrations in soil at isolated locations with Buildings 104 and 106 exceeded screening levels for human health. Stoddard solvent concentrations in the vicinity of Building 112A and the former Stoddard solvent underground storage tanks were detected at levels requiring further consideration by Alcoa. Removal of soil containing Stoddard solvent is not proposed as part of the below grade demolition and remediation.

Asbestos-containing materials (ACM), PCBs, and metals were detected in building materials at the Site. ACM (>1%) was detected in vinyl floor tiles and mastics, textured paints, HVAC sealant, corrugated wall panels (Galbestos panels), roofing mastic throughout the facility, and insulation debris on the roof of Building 104. PCBs were primarily detected in concrete floor samples from Building 104, on building surfaces, and in bulk samples of the Galbestos panels and wood block floors. Lead-based paint was identified throughout the facility. Metals were detected in the wipe samples collected from concrete floors, wood block floors, an I-beam flanges, wall trusses, and column footings.

Based on this information, the primary contaminants to be sampled during above grade structural demolition are PCBs and lead with the addition of PCE; TCE; benzene; 1,2,4-trimethylbenzene; and 1,3,5-trimethylbenzene during below grade demolition and/or soil



remediation. Benzene; 1,2,4-trimethylbenzene; 1,3,5-trimethybenzene will be used as indicators for Stoddard solvent emissions since these compounds represent some of the more toxic constituents and/or more prevalent constituents in Stoddard solvent. Asbestos is not included in this perimeter air sampling program since air monitoring and sampling will be the responsibility of the asbestos abatement contractor and abatement monitor (Aurora) during that phase of work. Although not required by SCAQMD, dust measured as particulate matter less than 10 microns (PM₁₀) will be measured to monitor compliance with SCAQMD Rule 403. Samples for PCBs; lead; PCE; TCE; benzene; 1,2,4-trimethylbenzene; and 1,3,5-trimethylbenzene will be collected to evaluate potential concentrations of these key contaminants in air dispersing from activities at the Site.

3.0 PROJECT DESCRIPTION

The planned activities at the Site consist of three general activities:

- Asbestos Abatement All air monitoring associated with asbestos removal will be conducted by the asbestos removal contractor and abatement monitor (Aurora) to meet regulatory requirements. Asbestos abatement is anticipated to take two months.
- Building Demolition Perimeter air monitoring during structural building demolition is addressed in this plan. Building demolition will consist of three phases: pressure washing of building surfaces, removal of aboveground structures, and removal of concrete pads and below grade structures. Perimeter air monitoring will be conducted during demolition of Buildings 104 and 106 and removal of concrete floor slabs from the same buildings, but will not be conducted during pressure washing inside structures. The construction manager will decide which specific activities and days will warrant sampling.
- Soil Remediation Perimeter air monitoring during soil remediation is anticipated to last approximately four month.

4.0 PERIMETER AIR SAMPLING

Perimeter air sampling will focus on these key potential emissions from the project activities during demolition:

- Dust measured as particulate matter less than 10 microns (PM10);
- Lead; and
- PCBs.



Perimeter air sampling will focus on these key potential emissions from the project activities during remediation:

- Dust measured as particulate matter less than 10 microns (PM10);
- Lead;
- PCBs;
- PCE,
- TCE.
- Benzene,
- 1,2,4-Trimethylbenzene, and
- 1,3,5-Trimethylbenzene.

Perimeter air sampling will be conducted to quantify airborne concentrations of PM-10, lead, PCBs, and VOCs (during soil remediation) at one upwind and two downwind locations during project activities. Wind direction and sampling locations were identified based on a windrose for Vernon, California for 1981 developed using data from the SCAQMD's website (http:\\www.aqmd.gov). As shown in the wind rose (Appendix A), the predominant wind direction is from west to east. Pre-designated upwind and downwind sampling locations along the western and eastern property boundary have been identified to make sample tracking easier. One upwind sampling location and two downwind locations will be monitored throughout the project. Two downwind locations will be located on the eastern boundary of the Site at least 200 feet apart when monitoring occurs. The upwind and downwind locations will move over the course of the project and will be placed in proximity to the most intense project work for that particular day. For example, building demolition is anticipated to move from north to south so the monitors would move to the pre-designated locations from north to south as demolition progressed. The designated sample locations and identifiers are shown on Figure 1. The upwind and downwind sample locations will have to be verified based on the actual wind direction on the day of the sampling. During remediation of VOC areas, an additional crosswind location will be monitored for ambient conditions based on the presence of an industrial cleaning facility in the vicinity of the site.

Perimeter air sampling data will be collected using air sampling devices followed by subsequent analytical laboratory analyses. Perimeter air samples will be collected using four



types of sampling devices. PM-10 air samples will be collected using PQ-100 or PQ-200 portable air samplers equipped with PM-10 inlets. The same sample collection devices and filters will be used for lead analysis. The PQ-100/200 samplers are approved by the EPA as portable samplers. The flow rate for the PQ-100/200 sampler is 16.7 liters per minute (lpm) and samples are collected on 47mm diameter Teflon filters. PCB samples will be collected using polyurethane foam cartridges (PUF) and a low flow sampling pump running at least 2.5 lpm. Volatile organic compounds will be collected using SummaTM canisters fitted with a flow control regulator.

Air samples will be collected daily during Site remediation work for the entire work day period (approximately 10 hours from 7 am to 5 pm) or longer if necessary. Air samples will only be collected over an entire work day since air samples collected for less than 10 hours may not achieve the detection limits necessary for the project. Samplers also will not be moved during the sampling period. At each air sampling station, the sampling devices will be set up with the air intakes elevated approximately 5 to 6 feet off the ground surface to collect a representative breathing zone sample. To the extent feasible, air samplers will be located away from large objects that may interfere with air movement near the sampler inlet. At the completion of the sampling period the sampling media will be uniquely labeled using the station identifiers, analyte, and the date. For example, identifier 1PCB-053106 would be used for a for a PCB sample at Station 1 on May 31, 2006. Abbreviations for analytes will be: PM10 for particulates, Pb for lead, PCB for PCBs, and VOC for VOCs. The samples will be individually packaged and shipped to an EPA accredited laboratory for analysis.

Detection limits in terms of air concentration will vary depending on how long the samplers are operated. PM-10 particulate weight will be determined gravimetrically by NIOSH Method 0500. Lead will be analyzed by NIOSH Method 7300. The expected limits of detection are 1 μ g/filter for lead and 50 μ g/filter for PM₁₀. Using the PQ-100/200 samplers at a flow rate of 16.7 lpm for a 10-hour workday, this equates to detection limits of 0.1 μ g/m³ for lead and 5 μ g/m³ for PM₁₀. PCB samples will be analyzed by EPA Method TO-10A. The expected limits of detection are 1 μ g/filter; using a low flow pump at a minimum flow rate of 2.5 lpm for a 10-hour workday, this equates to a detection limit of approximately 0.7 μ g/m³. VOC samples will be analyzed by EPA Method TO-15 using medium level reporting limits. The expected limits of detection will be less than 0.015 μ g/L for the VOCs.



Samples will be analyzed with a normal laboratory turn around time of 5 working days. Including time for sample shipment to the laboratory, sampling results will generally be available seven working days after sample collection.

Background air sampling will be conducted on three days prior to any dust-generating activities to evaluate background concentrations of PM-10 and lead in ambient air in and around the work area and to confirm sampling equipment is fully operational. Background air sampling will also be conducted on three days prior to demolition of concrete and excavation in areas of VOC-affected soil. A summary of the perimeter air sampling methods and action levels is shown in Table 1.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

A program of quality assurance and quality control (QA/QC) will be followed during implementation of this Plan to ensure consistent data collection and analysis procedures and to ensure that the data are representative of Site conditions. QA/QC procedures will be implemented to ensure correct operation of the monitoring/sampling equipment, and to validate the analytical data. The QA/QC procedures are discussed in the Quality Assurance Project Plan (QAPP, 2007b).

5.1 EQUIPMENT CALIBRATION AND MAINTENANCE

Manufacturer's specifications and operations manuals for each of the air monitoring devices to be used during the perimeter air sampling program are included in Appendix B. Calibration and maintenance procedures are summarized below.

5.1.1 Meteorological Monitoring Station

The Met One AutoMet sensors, datalogger, 3-meter stand, and solar power system will be set up and wired according to manufacturer's instructions. Wind direction, wind speed, temperature, relative humidity and barometric pressure will be data logged over 15 minute averaging periods for the duration of the work day. The data logged information will be downloaded at the end of each work day. Calibration of the sensors is done annually by the equipment rental company. The meteorologic monitoring station will be set up at the northeast corner of the site where power will be available but away for contractor activities.



5.1.2 **PQ-100/200** Air Sampler

The PQ-100 sampler will be assembled and programmed according to the manufacturer's manual. Target flow rates are pre-programmed and calibrated by the manufacturer, and recalibration should not be required. Flow rates will be verified on a daily basis using a DeltaCal or TriCal flow meter to check for air leaks.

PQ-100 samplers used for collection of airborne samples will be programmed with a target flow rate of 16.7 liters per minute. New 47mm Teflon filters will be placed in the filter holder each day. Filters placed in the PM_{10} samplers will be pre-weighed at the laboratory. Start time, stop time, and flow rates will be recorded in the daily field notes for each sampler.

5.2 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

A chain-of-custody (COC) will be prepared for each day's samples and will include the project number, sample date, sample numbers, sample volume, analyses requested, and the sampler's signature. Samples and the original COC will be shipped to the laboratory using an overnight courier service, with consideration of holding times and weekend sample receipts at a designated laboratory. Samples may also be picked up at the Site by the laboratory. Copies of the COC will be kept with the daily field notes. Holding times for air monitoring samples are as follows:

- PCBs PUF cartridge samples need to be extracted by the laboratory within 7 days of sample collection and analyzed within 40 days after sample extraction. Sample preservation includes storing samples in a chilled cooler (at 4 degrees Celsius).
- PM10 and Pb PQ-100/200 samplers have a 6 month holding time before analysis is required. No sample preservation is necessary.
- VOCs Summa canister with a regulator for 10-hour work day have a 30 day holding time from sample collection to analysis. No sample preservation is necessary.

For samples requiring temperature preservation, a temperature blank will be placed in the colder during along with samples. The temperature blank will be clearly marked as such.

5.3 QA/QC SAMPLES

A field blank consisting of unused filter media will be shipped to the laboratory along with other samples and analyzed to check for contamination during media preparation or field procedures. At least one field blank per month will be analyzed for each type of sampling media (i.e., filters, PUF samplers, and Summa canisters).



6.0 ACTION LEVELS

The action level for PM-10 particulates will be $50 \,\mu\text{g/m}^3$ based on the California ambient air quality standard and as specified in South Coast Air Quality Management District (SCAQMD) Rule 403. The action level for lead will be $0.3 \,\mu\text{g/m}^3$ based on the California Air Resources Board's Risk Management Guidelines for New, Modified, and Existing Sources of Lead (March 2001). This level is applied as a 30-day average, but for the purpose of this Plan will be used as an action level. This level is the acceptable concentration of lead in air for an area with average exposure to lead based on house age and income level as described in the guidelines.

The remaining action levels are based on minimum risk levels published by the Agency for Toxic Substances Disease Registry (ATSDR) for substances that are commonly found at Superfund sites or other regulatory screening criteria if MRLs were not available. A minimum risk level is an estimate of the daily human exposure to a hazardous substance that is likely to be without appreciable risk of adverse health effects over a specified duration of exposure-chronic, intermediate, and acute. Minimum risk levels are based on non-cancer health effects for the most sensitive health effects for the specific route of exposure. Minimum risk levels have been developed for acute (1 to 14 days), intermediate (15 to 365 days), and chronic exposure (more than 365 days). For this monitoring program for a duration of approximately 5 months, intermediate minimum risk levels will be used unless unavailable, in which case chronic minimum risk levels will be used.

The action level for PCE will be 0.27 micrograms per liter (μ g/L), the minimum risk level for chronic exposure (ATSDR, 2006) since an MRL for intermediate exposure has been published. The action level for TCE will be 0.54 μ g/L, the minimum risk level for intermediate exposure. The action level for benzene will be 0.029 μ g/L, the minimum risk level for intermediate exposure. Table 1 lists the action levels for this project.

An MRL for inhalation of 1,2,4-trimethylbenzene and 1,3,5-trimethylbenzene was not available. The action level for 1,2,4-trimethlbenzene and 1,3,5-trimethylbenzene will be 0.062 micrograms per cubic meter ($\mu g/m^3$) based on 10 times the preliminary remediation goal for chronic exposure published by the U.S. Environmental Protection Agency. The chronic exposure level is based on noncarcinogenic effects over a lifetime of exposure. For a short-term project such as this, the duration of exposure is significantly lower, corresponding to a higher allowable concentration in air.



An MRL for inhalation of PCBs was not available. The action level for PCBs will be $1.0 \,\mu\text{g/m}^3$ based on 10 times the preliminary remediation goal for chronic exposure published by the U.S. Environmental Protection Agency. The chronic exposure level is based on carcinogenic effects over a lifetime of exposure. For a short-term project such as this, the duration of exposure is significantly lower, corresponding to a higher allowable concentration in air.

If measurements exceed action levels, work will stop and additional dust (for dust, lead, or PCBs) or vapor controls (for VOCs) will be implemented. For dust, the following activities will be implemented:

- Apply water spray or mist or
- Slow work.

For VOCs that exceed action levels, the following activities will be implemented:

- Cover subject soil with clean soil;
- Slow work;
- Reduce size of area being excavated; and/or
- Apply vapor suppression.

Additional air monitoring may be conducted to confirm the effectiveness of emission reduction activities.

7.0 DOCUMENTATION AND REPORTING

A daily record of significant events and observations during the perimeter sampling will be recorded on daily field records. Periodic notation of meteorological measurements will be recorded on the Meteorological Monitoring Form. Periodic checks of the flow rate readings on the PQ-100/200 samplers will be recorded on the Air Sampling Forms.

A final written report of the Perimeter Air Monitoring Program will be prepared. This report will include a discussion of sampling methods and procedures, evaluation of the results, calibration and quality control information, and copies of field sampling forms and laboratory reports with chain-of-custody records.



8.0 REFERENCES

- Agency for Toxic Substance Disease Registry, 2006, Toxicological Profiles, http://www.atsdr.cdc.gov/toxpro2.html
- Geomatrix Consultants, Inc., 2006a, Below Grade Demolition Plan, Former Pechiney Cast Plate Facility, Vernon, California, December.
- Geomatrix Consultants, Inc., 2006b, Site Health and Safety Plan, Pechiney Cast Plate Facility, Vernon Facility, 3200 Fruitland Avenue, Vernon, California, July.
- Geomatrix Consultants, Inc., 2007a, Feasibility Study/Remedial Action Plan, Former Pechiney Cast Plate Facility, Vernon, California, July.
- Geomatrix Consultants, Inc., 2007b, Quality Assurance Project Plan, Former Pechiney Cast Plate Facility, Vernon, California, July.



TABLES



TABLE 1 PERIMETER AIR SAMPLING METHODS AND ACTION LEVELS

Pechiney Cast Plate Facility Vernon, California

Parameter and Equipment	Method (Method Detection Limit)	Frequency and Location	Estimated Number of Sampling Days	Action Levels
PM-10 Particulates PQ-100/200 sampler with PM-10 inlet using pre- weighed 47mm diameter Teflon filters.	NIOSH 0500 for particulate weight (50 µg/filter or about 6 µg/m³)	One upwind and two downwind locations ¹ at least once per week during building demolition ² and soil remediation.	3 background 1 per week for 5 months	50 μg/m ³
Lead PQ-100/200 samplers using 47mm diameter Teflon filters.	NIOSH 7300 for lead. (1 µg/filter or about 0.1 µg/m³)	One upwind and two downwind locations ¹ at least once per week during building demolition ² and soil remediation.	3 background 1 per week for 5 months	0.3 μg/m ³ for lead
PCBs Polyurethane foam (PUF) cartridge	TO-10A for PCBs (1 µg/cartridge or about 0.6 µg/m³)	One upwind and two downwind locations ¹ at least once per week during building demolition ² and soil remediation in PCB areas.	3 background 1 per week for 5 months	1.0 μg/m ³
VOCs Summa TM canister with regulator for 10-hour work day	TO-15 for tetrachloroethene, trichloroethene, benzene, 1,2,4-trimethylbenzene, and 1,3,5-trimethylbenzene (Approximately 0.002 µg/L)	One upwind, one crosswind, and two downwind ¹ locations at least once per week during soil remediation in VOC areas.	3 background 1 per week for 4 months	PCE -0.27 μg/L TCE -0.5 μg/L Benzene -0.029 μg/L 1,2,4-TMB -0.062 μg/L 1,3,5-TMB -0.062 μg/L

Notes:

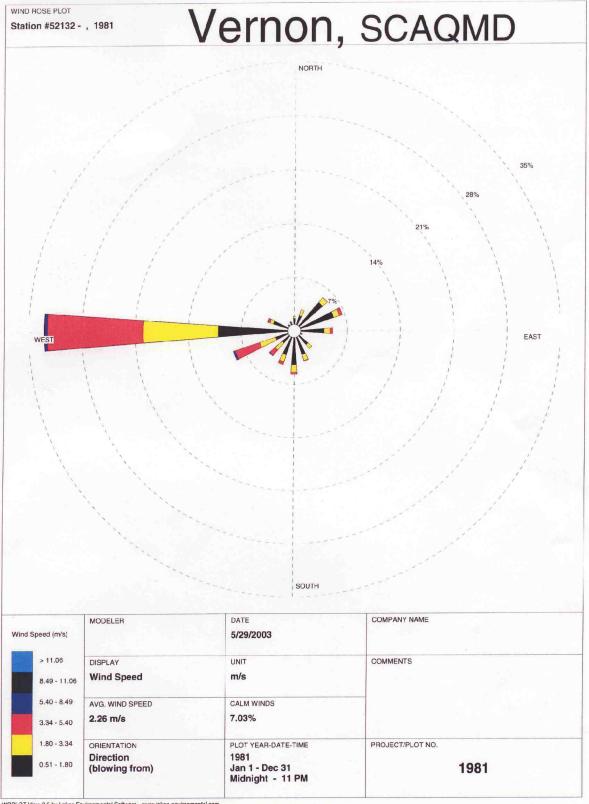
- 1. Upwind and downwind locations will be moved during Site activities to the western and eastern perimeters closest to actual field activities. Downwind samplers will be placed at least 200 feet apart.
- 2. Air samples will not be collected during power washing of interior surfaces.



FIGURES



APPENDIX A



WRPLOT View 3.5 by Lakes Environmental Software - www.lakes-environmental.com



APPENDIX D

PERTINENT FORMS

DAILY FIELD RECORD



							- 3	
Project and Tas	k Number:		Date:					
Project Name:				Field Activity:				
Location:				Weather:				
PERSONNEL:	Name		Company		Time In	Time Out		
	AFETY CHECKLIST					_		
	ed Boots	Hard Hat			Tyve	ek Coveralls		
	Gloves						-Face Respirator	
DRUM I.D.	ON OF C	CONTENTS	AND QUANTITY		LOCATIO	N		
TIME	DESCRIPTION OF WORK PERFORMED							
	<u> </u>							
	_							

DAILY FIELD RECORD (continued)



Page ____ of ____

Project and Tasl	k Number:	Date:
TIME	DESCRIPTI	ON OF WORK PERFORMED

NEW 11234

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RS (SIGNATURE):				LABORATORY PHONE NUMBER:		SITE SPECIFIC GLOBAL	ID NO.			
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	COMPANY:			COMPANY:	Tel 949.			,]))	

FIELD INSTRUMENT CALIBRATION SHEET

Project Name:	Project Number:	
Date:		
Equipment Type: Manufacturer:		
Model Number:		
Calibration (as necessary, minimum twice per day):		
Calibration #1	Time:	
Calibration Standard:		
Instrument Reading:		
Calibration #2	Time:	
Calibration Standard:		
Instrument Reading:		
Calibration #3	Time:	
Calibration Standard:		
Instrument Reading:		
Calibration #4	Time:	
Calibration Standard:		
Instrument Reading:		
Date of Last Calibration:	Date(s) Instrument Used:	
Name of person(s) who calibrated instruments:		
Calibration Standards Used:		
(1)		
(2)		
(3)		
(4)		
Source of Calibration Standards:		
Misc. Comments:		
	Calibrated by:	_

LAB REPORT QUALITY ASSURANCE CHECKLIST

Project Infor	matior	n					
Project Name:				Lab	Name:		
Project Number:			-	Lab	Report	Number:	
Sample Numbers: (Attach list if needed)							
Report Comp	oletene	ess					Comments
_		the COC included in			□ Yes		
report? (Indicate of resolve with the lab.		ences in Comments colur	nn and		□ No		
		sted on the COC for			□ Yes		
sample included in Comments column		eport? (Indicate any diff olve with the lab.)	ferences		□ No		
Are all items required by the contract with the included in the report? (Indicate any exceptions in a					□ Yes		
included in the re		Indicate any exceptions ii	n the		□ No		
QA Review of Lab Performance							Comments
Do all reporting limits meet project requiremed (Indicate any differences in Comments column and resonance)					Yes		
(Indicate any differences in Comments column and res with the lab.)			solve		No		
	sessme	nt Summary form			Attach	ed	
Inorganic Data Assessment Summary form					Attach	ed	
Field Blank ()A Re	view					
Are there any de	tections	s in the trip blanks?	□Ye	es	If yes, ic	lentify associate	ed samples:
			□ No)			
-	tections	in the equipment	□Ye	es	If yes, ic	lentify associate	ed samples:
blanks?			□ No)			
			•				
Invoice Revie	W						
Did the lab meet the promised turnaround			□Ye	□ Yes If no, d		es a discount a	pply?
times?			□ No				
Did any problems result in unusable			□Ye	es	If yes, evaluate whether the lab should be paid for		
sample results?			□ No		the analysis.		
Are all items required by the contract with			□Ye	es	If no, in	dicate any exce	ptions:
the lab included	in the re	eport?	□ No)			
Completed by:					Date:		
Reviewed by:					Date:		

INORGANIC DATA ASSESSMENT SUMMARY

Project Information	n							
Project Name:	Project Name: La			Lab Name:				
Project Number:			Lab Report Number:					
Reviewer's Signature:]		Samples:				
Review Date:			Matrix:					
Assessment Summ	ary							
Using the codes O, M, batch or sample deliver in the space provided.								
Meth	nod/Analyte:							
Other	descriptors:							
1. Preservation/hold tir	nes							
2. Calibration								
3. Blanks								
4. Interference check sample								
5. Lab control sample								
6. Duplicate sample analysis								
7. Spike sample analys	is							
8. ICP serial dilution								
9. ICP-MS tune analysis	is							
10. ICP-MS internal sta	andards							
11. Field duplicates								
12. Overall assessment								
Assessment Codes: O = No quality controls	s (QC) proble	ems were ide	entified for th	nese criteria				
M = The results are qua with a QC flag ind the method.			-			-		
Z = The results are una rejected (R).	cceptable due	e to gross Q	C problems.	The results	will be o	qualified as		
X = QC problems were	e identified, b	ut they do n	ot affect the 1	results, or th	ne review	ver is not		

certain of the effect on the results; or supporting documentation or data is not present in the

laboratory data package.

Assessment Code	Description	Action Required

ORGANIC DATA ASSESSMENT SUMMARY

Project Information	n								
Project Name:			La	b Name	:				
Project Number:			La	Lab Report Number:					
Reviewer's Signature:					Samp	oles:			
Review Date:			M	atrix:					
			•			•			
Assessment Summ	ary								
Using the codes O, M, batch or sample deliver in the space provided.									
Me	ethod Name:								
Meth	od Number:								
1. Preservation/hold tin	nes								
2. GC/MS tune, instr. p	erformance								
3. Calibrations									
4. Blanks									
5. Surrogates									
6. Matrix spike/dup									
7. Lab QC samples									
8. Internal standards									
9. Compound ID									
10. System performance	e								
11. Field duplicates									
12. Overall assessment									
Assessment Codes:									
O = No quality control	s (QC) proble	ems were i	denti	fied for t	these c	riteria.			
M = The results are qua with a QC flag ind the method.				-				-	
Z = The results are una rejected (R).	cceptable due	e to gross (QC pı	roblems.	The	results	will be o	qualified as	S
X = QC problems were	identified, b	ut they do	not a	ffect the	result	s, or th	e review	er is not	

certain of the effect on the results; or supporting documentation or data is not present in the

laboratory data package.	

Assessment Code	Description	Action Required